EFFECT OF PROCESSING ROUTES ON THE PROPERTIES OF THE STRIP MADE FROM HOGANAS SPONGE IRON POWDER

by

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by

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to the

DEPARTMENT OF METALLURGICAL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY, KANPUR

SEPTEMBER, 1986

To

the memory of my dear sister

CERTIFICATE

This is to certify that the present work on "Effect of Processing Routes on the Properties of the Strip Made from Hoganas Sponge Iron Powder," has been carried out by Mr. S.V. Ilango under my supervision and it has not been submitted elsewhere for a degree.

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S. V. Ilango

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SYNOPSIS

In the present work, an attempt has been made to compare the mechanical and structural properties of the strips obtained from sponge iron powder by two different powder metallurgical processing routes, viz. (i) powder preform → sintering → repeated cold rolling → sintering/annealing and (ii) powder preform → sintering → hot rolling → cold rolling → annealing.

It has been shown that the mechanical properties of the strip made from the above two routes are almost same. The amount of rolling deformation required to produce fully dense strip is similar, around 60-67% for both the routes. The volume percentage of inclusions present in the strip is nearly same in both the routes, but there is a marginal difference in the maximum size and number of inclusions in both the strips. The mechanical properties of the strip obtained in the present work are comparable with the strip obtained from other iron powders such as HVA electrolytic iron powder, sponge iron powder MH 100.24 But the properties are lower than those of the conventional low carbon mild steel strip.

CHAPTER I

INTRODUCTION

For most common metals, strip constitutes a major part of total 'product-mix'. For example, on a world wide scale flat sheet products including sheet and strip production are approximately 20% of the total, and the percentage is rising. Clearly the metal strip industry is operating on a large scale in financial terms, and therefore developments in the metal strip manufacturing technology is of great importance. Most thin metal strips are produced by static casting ingots from liquid metal followed by extensive hot and cold rolling. The thickness of the statically cast ingot for subsequent rolling is enormous compared with the thickness of the final strip, say 0.25 mm to 0.9 mm depending upon the condition. For example, the thickness of the statically cast steel ingot is about 500 mm, while 380 mm in the case of copper and copper alloys. The thickness reduction is so great that it causes a major increase in energy requirements which in turn increase the capital equipment and operating costs. Moreover the yield of finished metal strip or sheet is low, of the order of 70% for steel and 54% for stainless steels[1,2].

The above drawback can be overcome by starting with thinner slab. The last decade has seen the shift towards continuous casting which enables a thinner starting slab to be made, requiring less total mechanical working, to produce finished strip. The thickness of the continuously cast slab is 100 mm to 150 mm for steel[3]. The energy requirement for the production of metal strip through continuous casting is also relatively low. The yield is relatively high e.g. 80% for mild steel, 65% for stainless steel and 82% for copper and copper alloys[2,4]. Although there are several advantages of the continuous casting method over the ingot casting method, its introduction does not radically change the situation especially for steels, although serious efforts are being made to cast thin strip continuously.

During the last few years, there has been consistent research and developmental efforts to devise alternative methods for making metal strip requiring small total mechanical working. The strip making methods based on powder metallurgy are one of the most important among the alternative methods developed to date, and this method has been extensively reviewed by Dube[5].

1.1 Powder Metallurgy (P/M) Routes for Making Thin Steel Strip:

The main advantages of making metal strip by the P/M routes are as follows:

(1) The minimum plant capacity for making strip by P/M route is considerably less than that for the economically viable plant based on the conventional route. For example, in the case of steel, a 50,000-100,000 tonnes per year plant is possible for P/M routes as compared to > 1 million tonne per year for the conventional route. In the case of copper a 10,000 - 20,000 tonnes per year plant is feasible[6,7].

(2) The capital equipment cost per tonne of strip is about 90 percent as against ~70% for the conventional route[7].

(3) The product yield from the P/M plants is about 90% as against ~70% for the conventional plant[8].

(4) The amount of rolling deformation necessary to produce final finished strip is about 5:1.

(5) The number of unit operations involved is less, and the strip can be obtained from the powder on a continuous basis.

(6) The operating cost of a P/M plant for strip making is relatively low.

(7) The time required to install a P/M strip plant is relatively short, around 18 months[9].

(8) From the same plant various type of products such as cladded strip, dispersion strengthened strips etc. can also be produced.

1.1.1 P/M Routes Based on the Rolling of Cold Metal Powder:

This method consists of the following unit operations.

(1) Preparation of the green strip

(2) Sintering of the green strip

(3) Densification of the green strip

(4) Final cold rolling and annealing of the strip

The schematic flow chart of the above method is given in Fig. 1.1.

1.1.1.1 Preparation of the Green Strip:

It can be defined as a mechanically bonded metal powder formed into a strip which is relatively porous and brittle. This green strip can be made in two ways.

(1) Direct metal powder rolling

(2) Bonded metal powder rolling.

(a) Direct metal powder rolling:

The original and most obvious way for making green strip is by supplying cold metal powder into the roll gap of a powder rolling mill, where the powder is subjected to sufficient pressure to form a self supporting green strip. This method is generally known as direct metal powder rolling. The mill may be horizontal or vertical. The vertical mill is preferred because this avoids the difficulty of feeding loose powder into the roll gap.

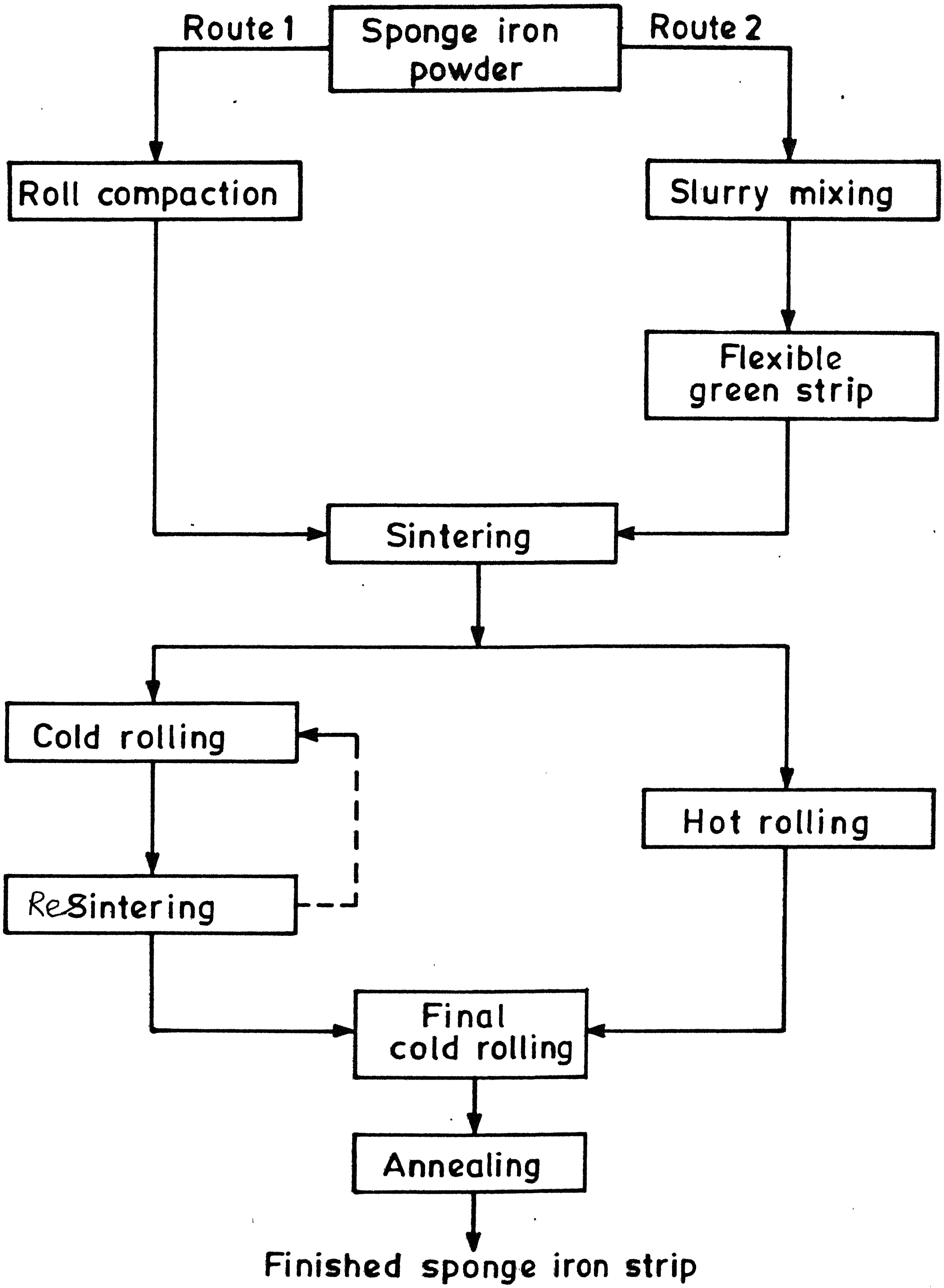


Fig. 1.1 Schematic flow diagram of P/M routes for making sponge iron strip.

Green strip formation into the roll bite can be divided into four zones.

(1) A free zone in which powder falls under gravity.

(2) A feed zone in which the powder is being dragged down by the rolls.

(3) A compaction zone, starting at nip angle or gripping angle, θ, within which the powder has become coherent.

(4) A deformation zone where deformation of powder takes place. It is believed that the green strength of the outcoming green strip is primarily developed in this zone.

The schematic diagram of different zones present in the roll bite has shown in the Fig. 1.2.

The entrainment of the powder into the compaction zone depends upon the feed angle, α, and the gripping angle θ. The feed angle depends upon the coefficient of friction μ, and any increase in μ increases the compaction zone.

The green strip properties of most interest are density, strength and thickness. These are functions of a) powder characteristics such as its flow properties, apparent density, shape, size and size distribution and b) mill parameters such as roll diameter, roll speed, roll gap and roll surface roughness.

The following are the problems associated with direct metal powder rolling.

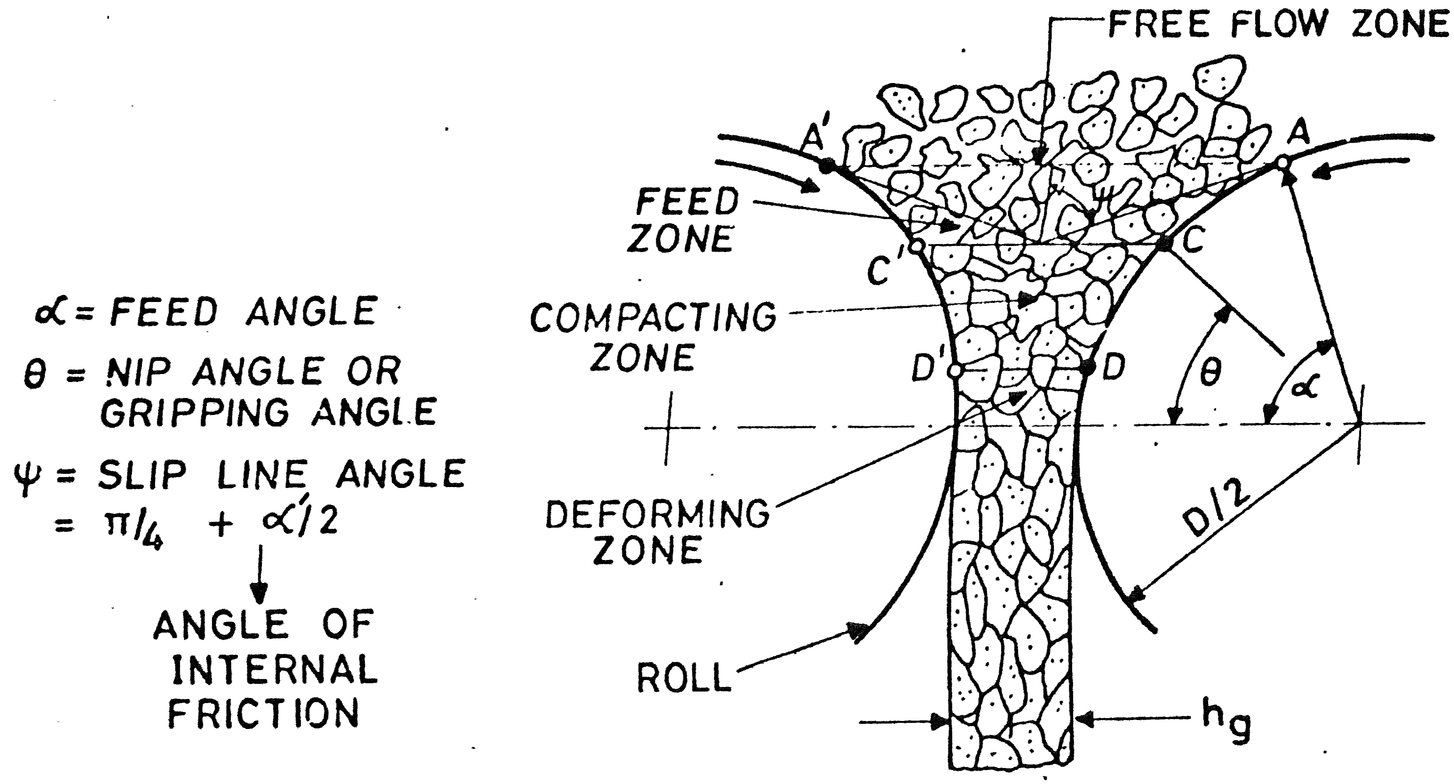


Fig. 1.2 Various zones within the roll bite during the direct cold metal powder rolling.

(1) Maximum thickness of the strip that can be produced from a given pair of rolls is severely limited.

(2) There is a limit to the actual speed of rolling. An increase above this limit does not give the expected increase in production rate owing to interference of the powder fed to the roll gap by the escape of the entrapped air. Although a speed as high as 0.5 m/s has been achieved for powder rolling, it is very low in comparison to the modern continuous mill where the speed is of the order of 16 m/s to 33 m/s.

(3) A major problem encountered in the direct metal powder rolling is the control of density distribution along the width of the strip[10]. Strips obtained have handling problems and low yield in the green state. This can be minimised by using edge restraints.

(b) Bonded metal powder rolling:

Some of the speed and metering problems experienced in the direct metal powder rolling can be overcome by feeding bonded metal powder in the form of a coherent strip into the roll gap. This enables the feeding of accurately metered quantities of powder into the rolling mill to be achieved. The preparation of green strip by this method consists of the following basic steps.

(1) Preparation of slurry mixture from metal powder, binder, plasticizer and solvent.

(2) Deposition of the slurry on a substrate.

(3) Drying of the slurry to form a coherent and flexible strip.

(4) Densification of the direct strip by roll compaction.

Here the thickness of the strip is independent of powder characteristics and mill parameters. However, the density of the green strip depends upon powder size, solid: liquid ratio in the slurry, viscosity of the slurry etc. The green strip after drying contracts to some extent depending upon the apparent density of the slurry.

Binder:

The purpose of the binder is to provide sufficient strength to green strip after the solvent has been evaporated. Binders are basically film formers[11]. A good binder should satisfy the following conditions.

(1) It should form a solution of sufficient viscosity at low concentrations and should be available in a wide range of viscosity.

(2) It should form a flexible self supporting film after drying.

(3) It must be readily removable during the sintering operations and should not leave undesirable residues.

(4) It should be relatively inexpensive.

Some of the commonly used binders are Methyl cellulose, Hydroxy propyl methyl cellulose, Hydroxy ethyl cellulose, Starches, Sodium Carboxymethyl cellulose, Sodium alginate and Ammonium alginate.

Solvent:

A solvent should have a low boiling point, low viscosity, ability to dissolve binder and plasticizer and non reactivity towards metal powder. Most of the non-aqueous solvents like acetone, benzene, butanol, toluene, ethanol etc. offer these advantages but suffer from disadvantages like high cost, high toxicity which give rise to potential inflammability hazards[12]. Water, on the other hand, is cheaper and non toxic but has a relatively higher boiling point.

Plasticizers:

Plasticizers are additives which soften the binder in the dry or near dry state. They are lower molecular weight organic compounds and dissolve in the same liquid as the binder. After drying, the binder and the plasticizer are intimately mixed as a single material. The plasticizer disrupts the close aligning and bonding of the binder molecules thereby increasing the flexibility of the material[13]. While softening the binder, plasticizers tend to reduce the strength of the bonded

powder preform. Some of the commonly used plasticizers are glycerine, polyethylene glycol etc.

The compositions of some typical slurries used for bonded cold metal powder rolling is given in Table 1.1.

1.1.1.2 Sintering:

Green strip obtained by either direct metal powder rolling or bonded metal powder rolling consists of mechanically bonded particles, and is relatively weak and brittle. It is therefore sintered to increase the strength. Simultaneously, sintering also removes undesirable impurities such as surface oxide films, sulphur etc. provided a suitable atmosphere is used.

Though it is possible to sinter coiled green strip[16,19], many practical problems such as danger of cracking of loose strip when bent and the possibility of edge damage might arise. Hunt and Eborall[16] sintered coiled green strip of copper at 1223 K. They observed that strip at 1223 K exhibited bad sticking and also considerable sagging. The strip sagged in such a way that the lower side became denser and developed a very marked curvature during subsequent rolling. It requires a considerable time to heat up and cool a large coil furnace. Additionally, there is a danger of sintering between the various layers of the coiled green strip.

Table 1.1: Composition of some typical slurries used for bonded cold metal powder rolling.

S.No.	Metal powder and its weight percent	Solvent and its weight percent	Binder and its weight percent	Plasticizer and its weight percent	Reference
1.	Iron, 70.0	Water, 29.4	Celacol, 0.6	None	Davies et al[8]
2.	Iron, 59.6	Water, 39.6	Celacol M450, 0.8	None	Dube[14]
3.	410SS, 80.0	Water, 19.7	Not mentioned,0.3	None	Wieland and Rudzki[15]
4.	430SS, 75.0	Water, 24.06	Not mentioned,0.4	Not mentioned, 0.50	Wieland and Rudzki[15]
5.	434SS, 80.0	Water, 19.35	Not mentioned,0.4	Not mentioned,0.25	Wieland and Rudzki[15]
6.	Iron, 87.8	Naptha, 11.47	Vistamex, 0.73	None	Davies et al[8]

Alternately, the green strip could be sintered in a horizontal sintering furnace which could be linked with the continuous green strip preparation unit. During the continuous sintering operation, the strip is supported on a mesh type belt made from heat resisting material. The continuous sintering, however, suffers from the disadvantage that the length of the furnace is determined by the sintering time required to produce sufficiently strong strip and the production speed of the strip. Therefore the main aim being the minimization of the furnace length. A high temperature - short time sintering is preferred. Many investigators[18-22] have now come to the conclusion that at temperatures between 0.7 and 0.85 of melting point of metal, sintering time may be reduced to a few minutes. Federchenko[23] et.al. found that the increase in temperature has a much greater effect then increasing the time, particularly at short sintering time.

While sintering the porous green strip, it is necessary to prevent its oxidation. Sometimes, oxides already present have to be reduced. It is always desirable that a reducing atmosphere is maintained in the sintering furnace. Dry hydrogen and endothermic gas are the common sintering atmospheres.

The sintering method proposed by Sakai[24] is important among the other alternative methods. In his method, the green strip is fed into a pair of graphite rolls carrying a low

frequency heavy alternating current (a few volts and 10,000 to 20,000 A), so that it is sintered instantaneously by the resistant heat produced within the strip. The interesting result was that the apparent density of the sintered strip was greater than that of the green strip and could be maximised by the proper selection of rolling speed. The optimum value of current density to produce maximum density in the sintered strip was, in general, 2 to 4 A/mm^2. It has been claimed that this method of sintering has considerable prospects although various problems still have to be resolved. More recently, Strugeon[25] has proposed that the green metal strip could be supported on the gas cushion during its movement in the sintering furnace. Such a sintering method, when adequately developed, eliminates the installation of conveyer belt required for the movement and support for the strip and offers advantages of good heat transfer and also the mass transfer. This method of sintering can also be used to reduce the oxides and to remove the sulphur, if adequately developed.

1.1.1.3 Densification Rolling of the Sintered Strip:

The strip obtained after sintering is still relatively porous, although its strength is greatly increased in relation to the green strip. The densification of the sintered strip into a fully dense strip can be carried out in two different ways.

(1) Repeated cold rolling and sintering/annealing cycle.

(2) Hot rolling.

(a) Repeated Cold Rolling and Sintering/Annealing Cycle:

Many workers[8,10,16,17,22] have reported that fully dense metal strip can be made from roll compacted and sintered strip by a series of cold rolling and sintering or annealing cycle. For example, Worn et al[27] in the case of nickel, Sturgeon et al[28] in the case of steel and Hunt and Eborall[16] and Katrus[22] et al in the case of copper found that fully dense strips from roll compacted and sintered strips containing 15 percent porosity can be obtained by this method when the total thickness deformation by cold rolling was approximately 70 percent. Worn et al[26] also observed that the initial cold rolling must be limited to ~20% thickness deformation, owing to the presence of highly dispersed porosity which makes the strip crack easily. However, subsequent reduction could be greater. Kimura[28] et al used the following procedure to make fully dense strip from the roll compacted and sintered iron strip having relative density about 90 to 95 percent of the theoretical on a laboratory scale.

10 percent thickness reduction by cold rolling

→ Sintering at 1373 K for 3600 Sec in dry hydrogen

→ 30 percent thickness reduction by cold rolling.

Neitzel et al[29] obtained iron and Cr-Ni steel strip having mechanical properties similar to those of conventional strip by

sintering the respective green strip at 1573 K to a density of 6.5×10^3 kg/m^3 followed by atleast 50 percent cold rolling and subsequent annealing at 1373 or 1423 K.

The amount of cold rolling required to achieve full density largely depends on the properties of the strip, particularly porosity and generally several cold rolling and sintering or annealing cycles are required. A further disadvantage is encountered when relatively thick wrought strip is required. If the cold rolling and sintering or annealing cycles do not produce full density until an overall reduction of 60 percent or more of the green strip thickness has been taken, a very thick green strip would be required as a starting material.

(b) Hot Rolling:

Many investigators[29,6,20,31-34,9] have favoured hot rolling after sintering as a means of achieving 100 percent density in the strip. The main advantage of this process is that it is possible to produce a dense strip in one operation, and it also reduces the sintering time, and hence the furnace length. Another interesting point is that it is possible to hot roll the sintered strip from the sintering furnace directly into a dense strip without cooling. The amount of thickness deformation required to produce a fully dense strip depends on the initial porosity and is always greater than that of the theoretically required. This is because of the fact that some

of the rolling deformation goes into elongating the strip instead of closing the pores.

Weaver et al[31] found that for sintered iron strip having relative densities of 39%, 62% and 77.5 % of the theoretical, a thickness deformation of about 70%, 45%and 35% respectively was required to achieve full density by hot rolling at 1313 K.

The results of Kimura[34] et al on iron powder showed that the properties of the final finished iron strip obtained from electrolytic iron powder by the P/M method involving hot rolling as a means of further densification compared favourably with those of the iron strip obtained from the same powder but made by the P/M method involving repeated cold rolling and sintering or annealing cycles as a means of densification. However, in the case of a sponge iron powder, they found the strip made from the former route was slightly inferior to the latter, because of its inferior sinterability.

The fully dense hot rolled strip could be either cooled in a protective atmosphere to give an oxide free bright surface[29] or could be partially cooled in a protective atmosphere followed by water cooling and pickling[6]. Very slow hot rolling would cause the steel rolls of the hot mill to overheat, which would result in a poor hot roll life. If these rolls are externally cooled while hot rolling slowly, the thin sintered strip will be quenched and proper hot rolling may not be achieved. It is

possible to keep the rolls below the tempering temperature without excessive cooling of the strip if hot rolling is done at higher speeds. The hot rolled strip should remain in a reducing atmosphere right upto the entry of the rolls to prevent internal oxidation of the strip resulting in poor properties.

1.1.1.4 Final Cold Rolling and Annealing:

The final cold rolling and annealing is done to achieve optimum mechanical and structural properties in addition to superior surface finish. P/M metal strip produced by a hot rolling method would have an inferior surface finish because of oxide film formation. Therefore it is necessary to pickle the sheet or strip produced from the repeated cold rolling and sintering or annealing method, This final cold rolling and annealing can be combined with the cold rolling and sintering or annealing carried out for densification purposes. The final annealing temperature and time for the P/M strip is, generally, greater than that used in the conventional processes and it has been found to be beneficial in achieving optimum properties[34]

It is desirable to start cold rolling on a fully dense P/M metal sheet strip. However, according to Smucker[6], the metal strips within the 96% to 100% relative density range can be cold rolled like a conventional 100% dense metal strip. Therefore the final cold rolling and annealing could be used for some densification. Nevertheless, the primary aim remains to achieve optimum mechanical properties and structural properties.

1.2 Aim of the Present Investigation:

The objectives of the present investigation were as follows.

(1) To study the effect of processing routes on the mechanical and structural properties of the strip obtained from Hoaganas sponge iron powder.

(2) To optimise the percentage cold rolling reduction and percentage hot rolling reduction required to make fully dense sponge iron strip.

(3) To compare the properties of the P/M strip made from Hoaganas sponge iron powder with conventional strip.

CHAPTER II

RAW MATERIALS AND EXPERIMENTAL PROCEDURE

2.1 Raw Materials:

2.1.1 Sponge Iron Powder:

Sponge iron powder having trade number NC 100.24 produced by Hoganas Limited, Swedan was used in the present investigations. The chemical analysis of the powder is given in Table 2.1. Powder of -100 mesh size was used to make the green strip.

2.1.2 Binder:

Reagent grade methyl cellulose was used as hinder. This was necessary to form a homogeneous slurry of the powder with sufficient viscosity.

2.1.3 Plasticizer:

Methyl cellulose, when used as a binder, produced green strip having a curled surface. In general, the green strip was hard. Glycerine was used to produce plasticizing effect in a methyl cellulose-water system. Reagent grade glycerine was used as plasticizer in the present investigation.

2.1.4 Gases:

Nitrogen gas was used to purge out the oxygen present inside the furnace before passing hydrogen at high temperature.

Table 2.1: Chemical Analysis of the NC 100.24 Sponge Iron Powder

Constituent	Weight percent
H_2 loss	0.30
C	0.02
SiO_2	0.26
P	0.015
S	0.015

Hydrogen gas was used to protect the green strip from oxidation and also to reduce the oxides present in the sample. IOLAR-1 grade hydrogen and standard nitrogen supplied in cylinders by Indian Oxygen Limited were used.

2.2 Experimental Procedure:

2.2.1 Preparation of Green Sponge Iron Strip:

Sponge iron powder and binder were weighed accurately and blended manually. This mixture was transferred to 400 ml beaker. Water was mixed with glycerine in a 100 ml beaker separately. Water-glycerine mixture was slowly added to sponge iron powder-binder mixture in a 400 ml beaker and blended thoroughly by using a power driven stirrer. The slurry thus formed contained some air bubbles which resulted into small blow holes in the green strip during mixing. Care was taken to minimize the trapping of air bubbles by using a lower speed of mixing. The composition of the slurry is shown in Table 2.2.

The free flowing slurry was poured into a rectangular mould of dimension 100 x 75 x 5 mm. Before pouring the slurry the mould surface was coated with oleic acid which act as a releasing agent. The cast slurry was dried on a hot plate.

2.2.2 Compaction of the Green Strip:

The dried green strip was pressed between flat platens on a hydraulic press using 300 N/mm^2 pressure. The compacted strip was found to have fragile edges because of the constraints from the sides, which were removed. The apparent density of the pressed strip was about 5 Mg/m^3.

Table 2.2

Composition of the slurry made in the present work.

Constituent	Weight %
Sponge Iron Powder	69.5
Methyl Cellulose	1.0
Glycerine	2.0
Water	27.5

2.2.3 Sintering of the Green Strip:

The sintering furnace used in the present investigation is shown in Fig. 2.1. The chamber of the furnace was 750 mm long and 100 mm in internal diameter. It was made up of Inconel tube. The chamber was closed at one end. The open end of the furnace had a 200 mm long cooling chamber where the sintered sponge iron strips were cooled to 70°C under hydrogen atmosphere prior to taking out from the furnace. Gases were introduced in the chamber through a 6 mm internal diameter stainless steel tube passing through the open end of the chamber and were released near its closed end. The unused reducing gas was burnt at the exit.

The standard procedure for sintering was that, the chamber was flushed with nitrogen for about 5 minutes before introducing nitrogen, the furnace was maintained at the required temperature. The green strip, placed in a perforated Inconel tray, was then pushed into the hot zone of the furnace. After the required sintering time, the tray was removed from the hot zone and placed at the cooling zone for 30 minutes before taking it out. In the present investigation, the green strip was sintered at 1150°C for 30 minutes. This temperature was selected because it is the maximum sintering temperature at which commercial production is carried out at minimum maintenance cost.

2.2.4 Densification Rolling of the Sintered Strip:

The Sponge iron strips obtained after sintering contained about 38 percent porosity. In order to obtain fully dense strips,

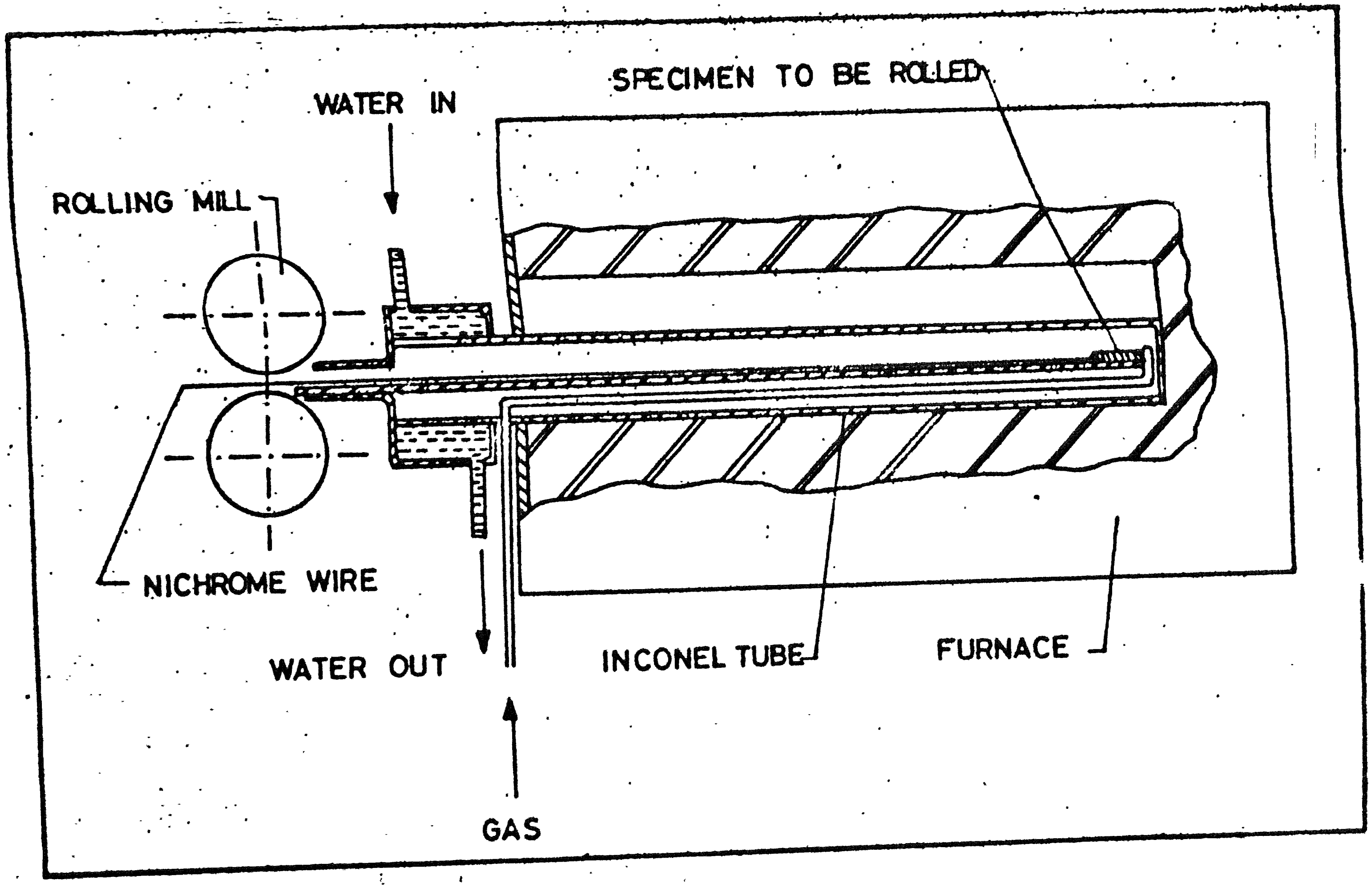


Fig. 2.1 Hot rolling arrangement.

it was necessary to densify them by rolling. The densification of the sponge iron strip was carried out by two different routes.

(a) Cold rolling - Sintering/annealing cycle.

(b) Hot rolling.

2.2.4.1 Cold Rolling - Sintering/Annealing Cycle:

The sintered and cooled strip was cold rolled on a two high mill having 135 mm diameter rolls and rotating at a speed of 55 rp In order to study the cold rolling behaviour of the sintered strip the strips were rolled to various thickness deformation, viz. 10, 20, 30, 40, 50, 60, 70 percent etc. and the mechanical properties were measured in as rolled condition and in as rolled and annealed condition.

It was found that the strips can not be cold rolled without rupture beyond 40 percent thickness reduction. Therefore, in all the cases of cold rolling beyond 40 percent, the strip was first rolled to 30percent thickness reduction followed by annealing at 1423K for 30 minutes. The remaining cold rolling reduction was then given to the strip.

2.2.4.2. Hot Rolling:

The schematic sketch of the reheating furnace and hot rolling operation is given in the Fig. 2.1. The reheating and hot rolling was done in hydrogen atmosphere to prevent oxidation of the strip during heating and also to reduce the surface oxides which are formed during storage. One end of the reheating furnace

was closed, while the other end had a zone projecting outside the furnace. The reheating chamber contained a flat plate of Inconel as a base for the strip. The sponge iron strip was preheated to 1423 K for 1800 secs. prior to hot rolling. The hot rolling was done on a 2-high mill having 135 mm diameter rolls rotating at a speed of 55 rpm. The standard procedure for hot rolling was as follows:

(a) A small hole was drilled near one edge of the porous strip and a thin nichrome wire was attached to it.

(b) The porous strip tied with nichrome wire was placed on an Inconel flat plate and introduced into the hot zone of the pre-heating furnace.

(c) The preheating furnace was moved to front side of the rolling mill, so that the extended exit end was very close to the nip of the rolls.

(d) The roll gap was adjusted to the required level and then the heated sponge iron strip was pulled into the rotating rolls from the hot zone with the help of the attached wire.

(e) The strips coming out of the mill were cooled in a bed of graphite chips for 5 minutes.

The hot rolled strips were annealed at 973 K for 2400 Secs. in hydrogen atmosphere to remove the work hardening introduced on the surface of the strip due to chilling caused by the relatively cold rolls.

2.2.5 Cold Rolling and Annealing:

The fully dense hot rolled strips were cold rolled to various thickness reduction followed by annealing at 700°C for 135 minutes in hydrogen atmosphere.

In the case of strips produced by repeated cold rolling-annealing/sintering route, the final annealing was done at 700°C for 135 minutes in hydrogen atmosphere.

2.3 Methods of Testing and Inspection:

2.3.1 Density:

The density of the porous strip was measured by weight and dimension method. The density of the fully dense strip was measured by using Archimedies principle.

2.3.2 Mechanical Properties:

Ultimate tensile strength and elongation were determined by a universal testing machine instron 1150. The testing was done at room temperature and samples were strained at a rate of 0.5 mm/minute. Because of the shortage of material, the size of the specimen used for mechanical testing was not according to the BS 18 specification. But the geometry of the specimen, as shown in Fig. 2.2 was maintained according to the standard specification. The elongation of the strips were calculated by measuring the distance between the markings made before testing by using a travelling microscope. A minimum of three specimen was tested in all the cases.

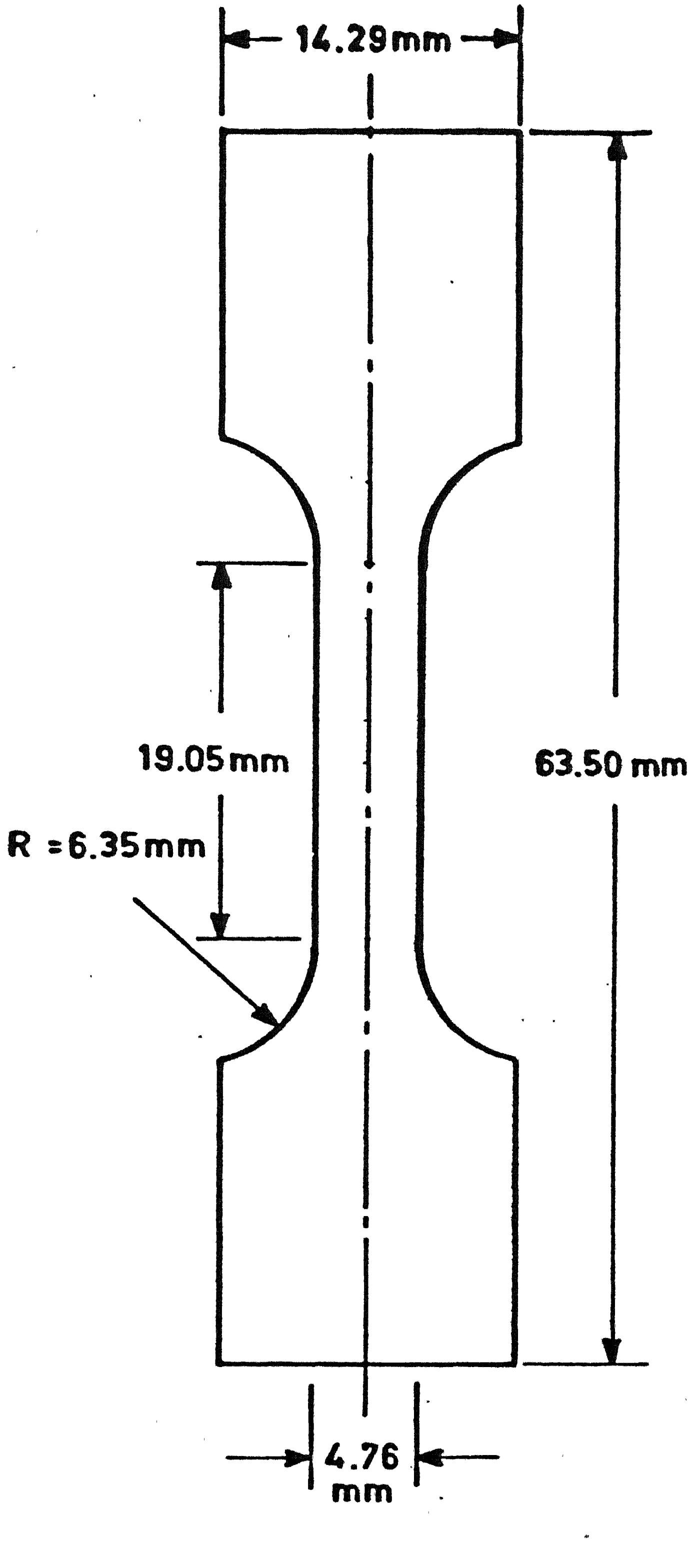


Fig. 2.2 Tensile test specimen.

2.3.3 Quantitative Analysis of Inclusions:

The following quantitative parameters related to the inclusions present in the strips were determined by using Image Analyser.

(1) Volume percentage of the inclusions.

(2) Size of the inclusions.

By using the image analyser, the total area of the frame and the total area of the inclusions present in the frame were measured in $(\text{micron})^2$ which was converted into percentage. The area of the inclusion was assumed to be equal to the volume percentage. The length of the inclusion was measured after calibrating the instrument by using the standard circle of diameter 100 micron at 400 x magnification. 30-40 measurements were taken and the data was statistically analysed. The standard procedure for calculating 95 percent confidence limit was adopted.

2.3.4 Metallography:

2.3.4.1 Optical Microscopy:

The mounting of porous strips for metallography consisted of impregnating it with araldite epoxy resin CY212 and Hardner - HY 951 mixture in the ratio of 10:1 by weight and subsequently curing it at room temperature. The standard polishing procedure was adopted. 2 percent nital solution was used for etching.

2.3.5 Scanning Electron Microscopy:

The fractured surface of the partially densified strip was examined under scanning electron microscope.

CHAPTER III

RESULTS AND DISCUSSION

The pressed compact, which had density of 5 gm/cm^3 was densified by

(1) Cold rolling - sintering/annealing cycle.

(2) Hot rolling.

3.1 Densification of Green Sponge Iron Strip by Cold Rolling - Sintering/Annealing Cycle:

3.1.1 Effect of Percentage Cold Rolling Reduction on the Density of the Strip:

Variation of density of the strip with percentage cold rolling reduction is shown in Fig. 3.1 and Table 3.1. It can be seen that as the percentage cold rolling reduction increases, the density increases linearly till it reaches the limit, after which no further increase in density was observed and it can be assumed that full densification in the strip has taken place. The full density of the sponge iron strip was observed at about 65% cold rolling.

3.1.2 Effect of Percentage Cold Rolling Reduction on the Mechanical Properties of the Strip:

The effect of percentage cold rolling reduction on the UTS of the strip, measured without annealing after cold rolling, is shown in Fig. 3.2 and Table 3.2. It can be seen that as the percentage cold rolling reduction increases, the UTS increases

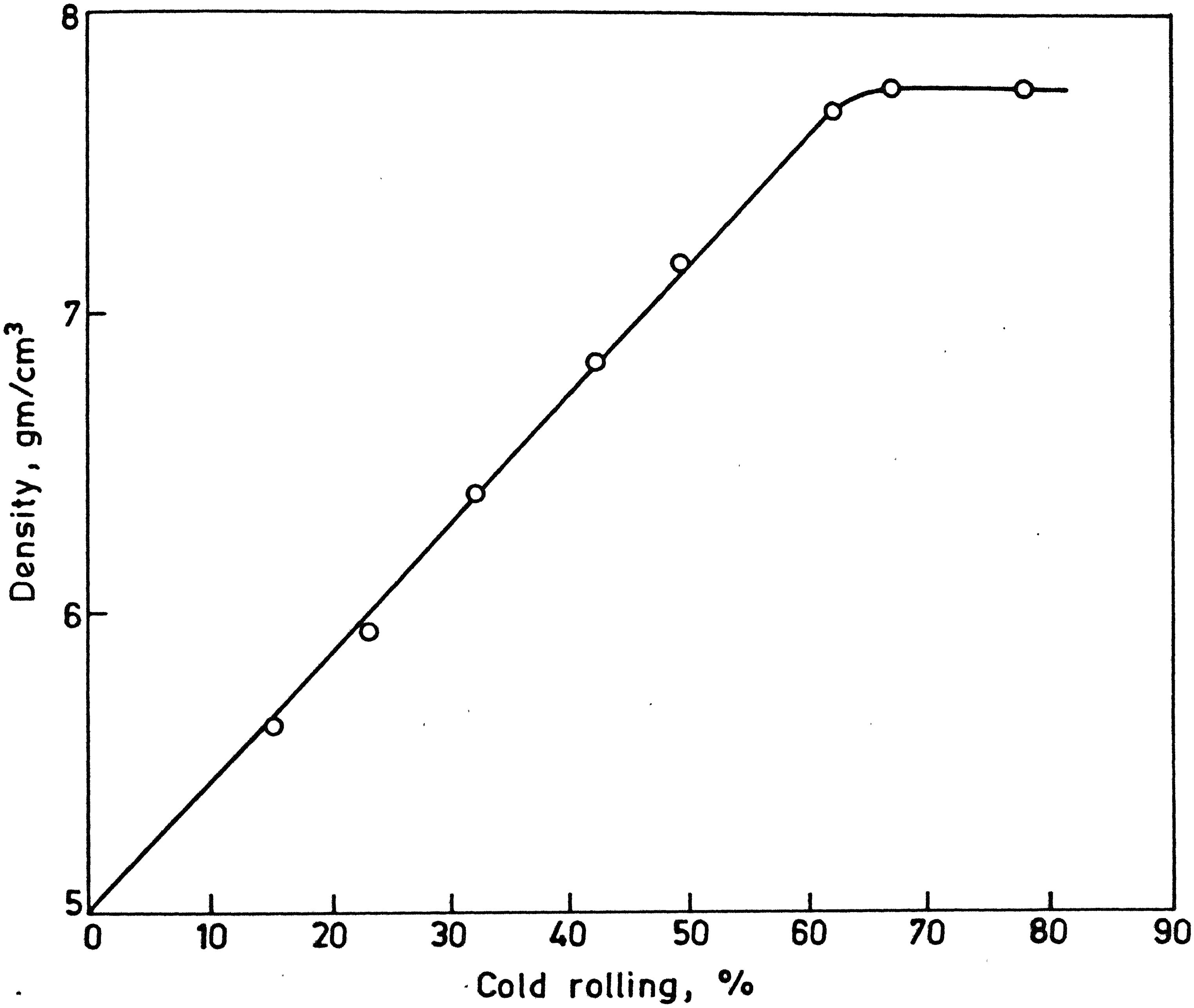


Fig. 3.1 Effect of percentage cold rolling reduction on the density of the sponge iron strip.

Table 3.1

Effect of percentage cold rolling reduction on the density of the sponge iron strip.

S. No.	Cold Rolling (%)	Density gm/cm^3
1	15	5.62
2	23	5.94
3	32	6.40
4	49	7.17
5	62	7.68
6	67	7.76
7	78	7.76

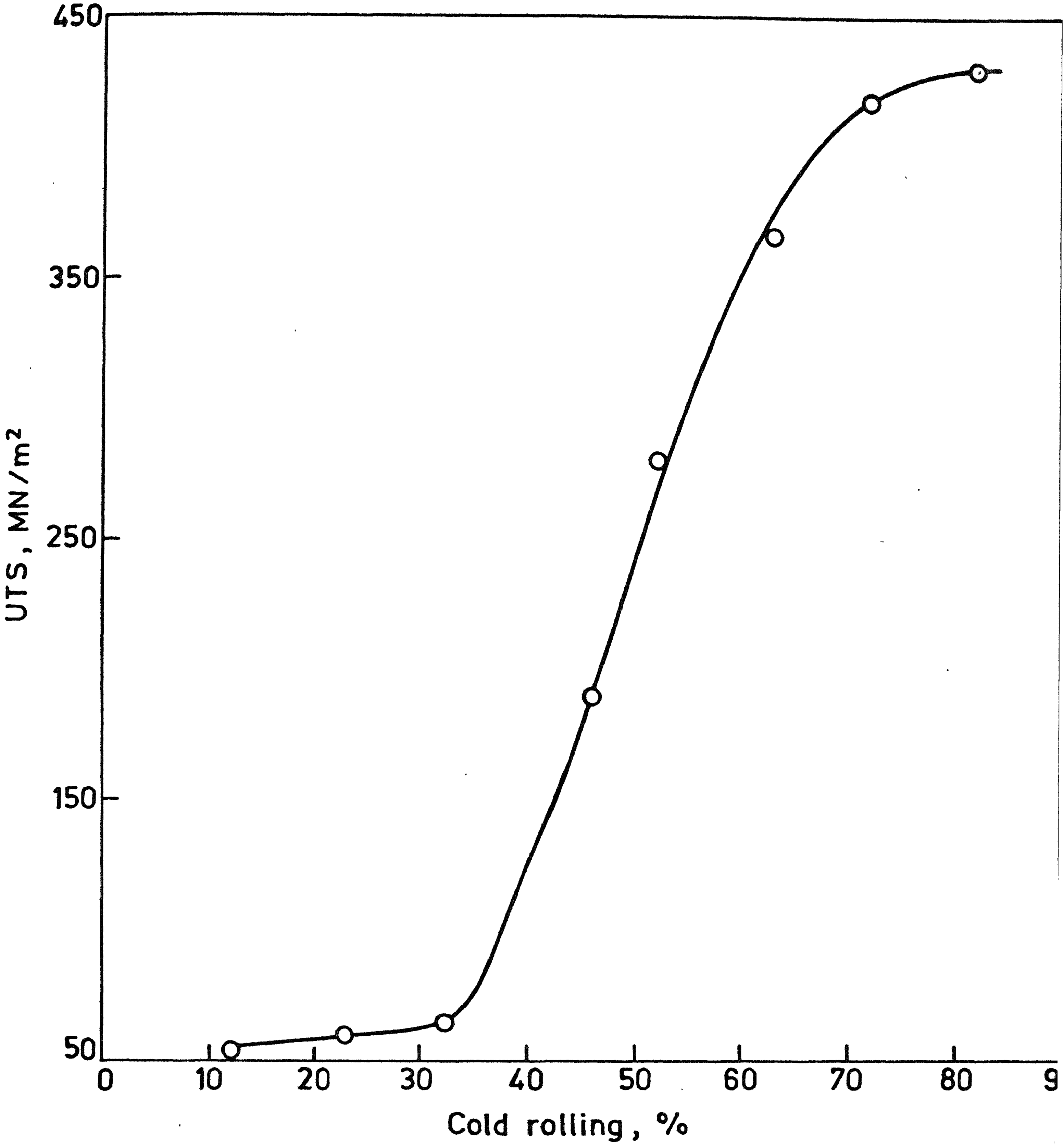


Fig. 3.2 Effect of percentage cold rolling reduction on the UTS of the cold rolled sponge iron strip.

Fig. 3.2

Effect of percentage cold rolling on the UTS of the cold rolled sponge iron strip.

S.No.	Cold Rolling (%)	UTS (MN/m^2)
1	12	54
2	23	60
3	35	64
4	46	190
5	52	280
6	63	366
7	72	418
8	82	430

at a slow rate till 32% cold rolling reduction and then increases rapidly upto 70% . Beyond 70% cold rolling reduction, the UTS remained constant. The elongation values of the specimen, as expected, were very low and could not be measured using standard procedure.

The effect of % cold rolling reduction on the UTS and % elongation of the strip, measured after annealing at 973 K for 8100 secs. in hydrogen atmosphere is shown in Fig. 3.3 and Table 3.3. The annealing treatment would remove the work hardening effect which was introduced during cold rolling. It can be seen that the UTS and % elongation increased at a slow rate till 45% cold rolling. After 45% cold rolling both the properties increased rapidly and reached the maximum at about 67% cold rolling reduction at which full densification has occured. However, a decrease in UTS and % elongation beyond 67% cold rolling reduction was observed. This behaviour is unexpected.

3.1.3 Effect of Percentage Cold Rolling on the Densification Efficiency of the Strip:

The efficiency of densification of the strip by cold rolling, E, was calculated by using the relation given below:

$$E = \frac{(\rho_{CR} - \rho_o)}{(\rho - \rho_o)} \times 100$$

ρ_{CR} - Density of sponge iron strip after cold rolling.

ρ_o - Density of the ~~initial~~ compacted sponge iron strip.

ρ - Density of the fully dense strip.

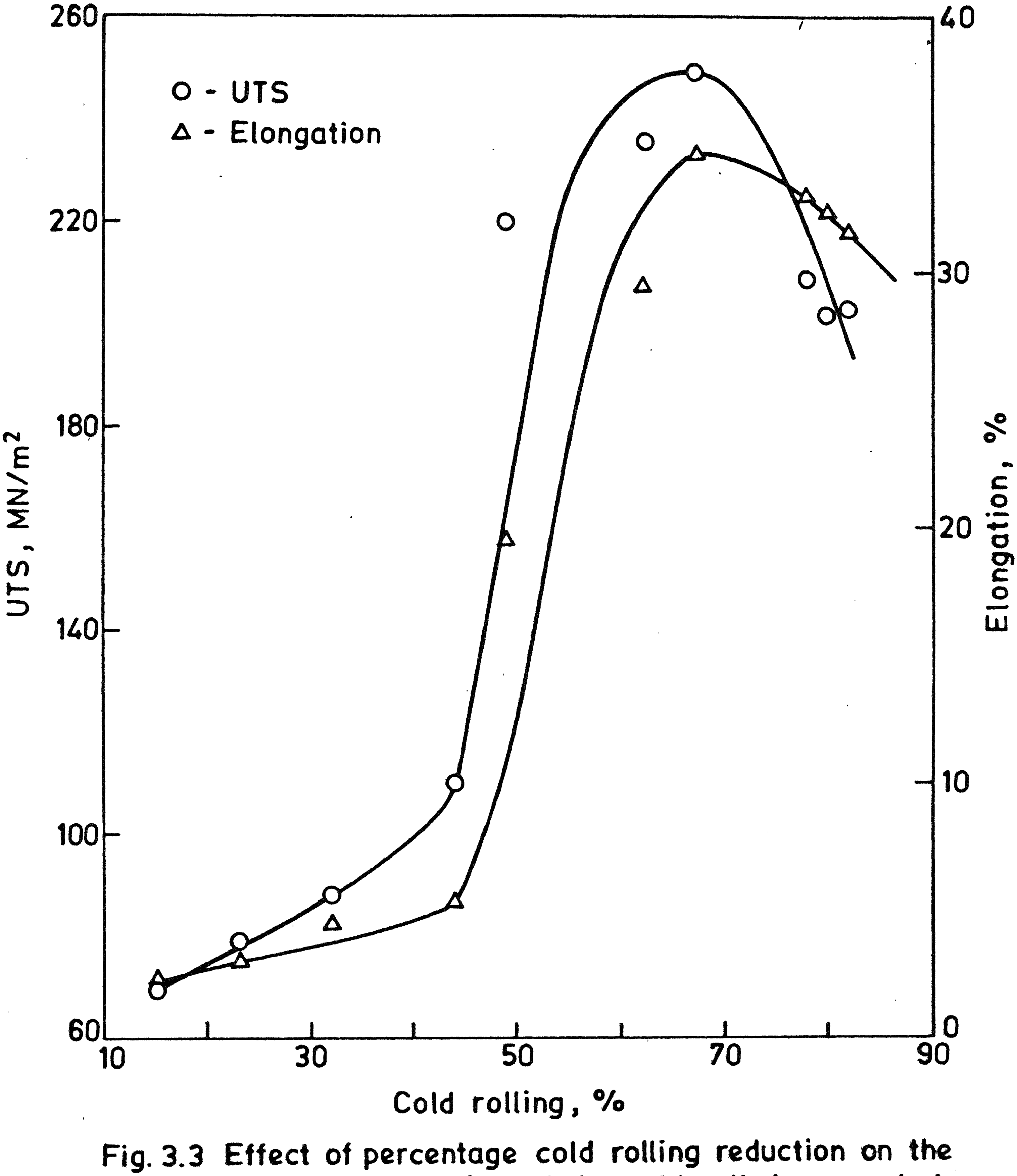


Fig. 3.3 Effect of percentage cold rolling reduction on the UTS and elongation of the cold rolled - annealed sponge iron strip.

Table 3.3

Effect of percentage cold rolling reduction on the UTS and elongation of the cold rolled-annealed sponge iron strip.

S.No.	Cold Rolling (%)	UTS MN/m^2	Elongation (%)
1	15	70	2.2
2	23	79	3.0
3	32	88	4.5
4	44	110	5.3
5	49	220	19.5
6	62	236	29.5
7	67	250	34.7
8	78	209	33.0
9	80	202	32.3
10	82	203	31.5

The density of mild steel or pure iron should not be taken as the density of the fully dense strip in the above equation, because of different chemical composition of the materials. The strip produced from sponge iron powder would contain some low density non-metallic inclusions which had been originated from the starting iron ore. The density of such a material would be lower than that of pure iron.

Fig. 3.1 shows that the density increases linearly and it reaches the maximum at 67% cold rolling reduction. After 67% cold rolling reduction, the density remains constant at a value of 7.76 gm/cm^3, suggesting that full densification has taken place. It can be concluded that the maximum achievable density is 7.76 gm/cm^3 for the sponge iron powder used. This value was taken as the theoretical density of the fully dense strip for densification efficiency calculation in the present work.

The theoretical density of a sponge iron strip can also be calculated from the chemical analysis of the powder and the density of the various ingredients. Such an exercise was carried out by Lindskog et.al.[41] on a similar Hoaganas sponge iron powder number SC 100.26 and the theoretical density value reported has been 7.75 gm/cm^3, which agrees with our assumed value.

Fig. 3.4 and Table 3.4 shows the effect of % cold rolling reduction on the densification efficiency of the sponge iron strip. As in the case of density, the densification efficiency also increases linearly with % cold rolling reduction, till it reaches the maximum, after which it remains constant. The maximum densification efficiency was obtained at 67% cold rolling reduction.

3.1.4 Effect of True Thickness Strain on the True Length Strain of the Strip:

Fig. 3.5 and Table 3.5 shows the relationship between true length strain and true thickness strain of the cold rolled sponge iron strip. The dotted line is for a fully dense material in which the true thickness strain would be equal to true length strain under plane strain condition. It can be seen that the true thickness strain changes almost linearly with the true length strain till the true thickness strain, at which full density occurs, is reached. Beyond true thickness of - 1.1 which corresponds to 65% thickness reduction, the length strain was equal to the thickness strain and the straight line become almost parallel to the dotted line for fully dense material.

3.1.5 Volume Percentage and Size Distribution of Inclusions in the Strip:

The volume fraction of inclusions present in the sample alongwith 95% confidence limit was found to be 2.87 $\pm$ 0.42 per-cent. The Figs. 3.6 and 3.14 and Table 3.6 and 3.7 shows the

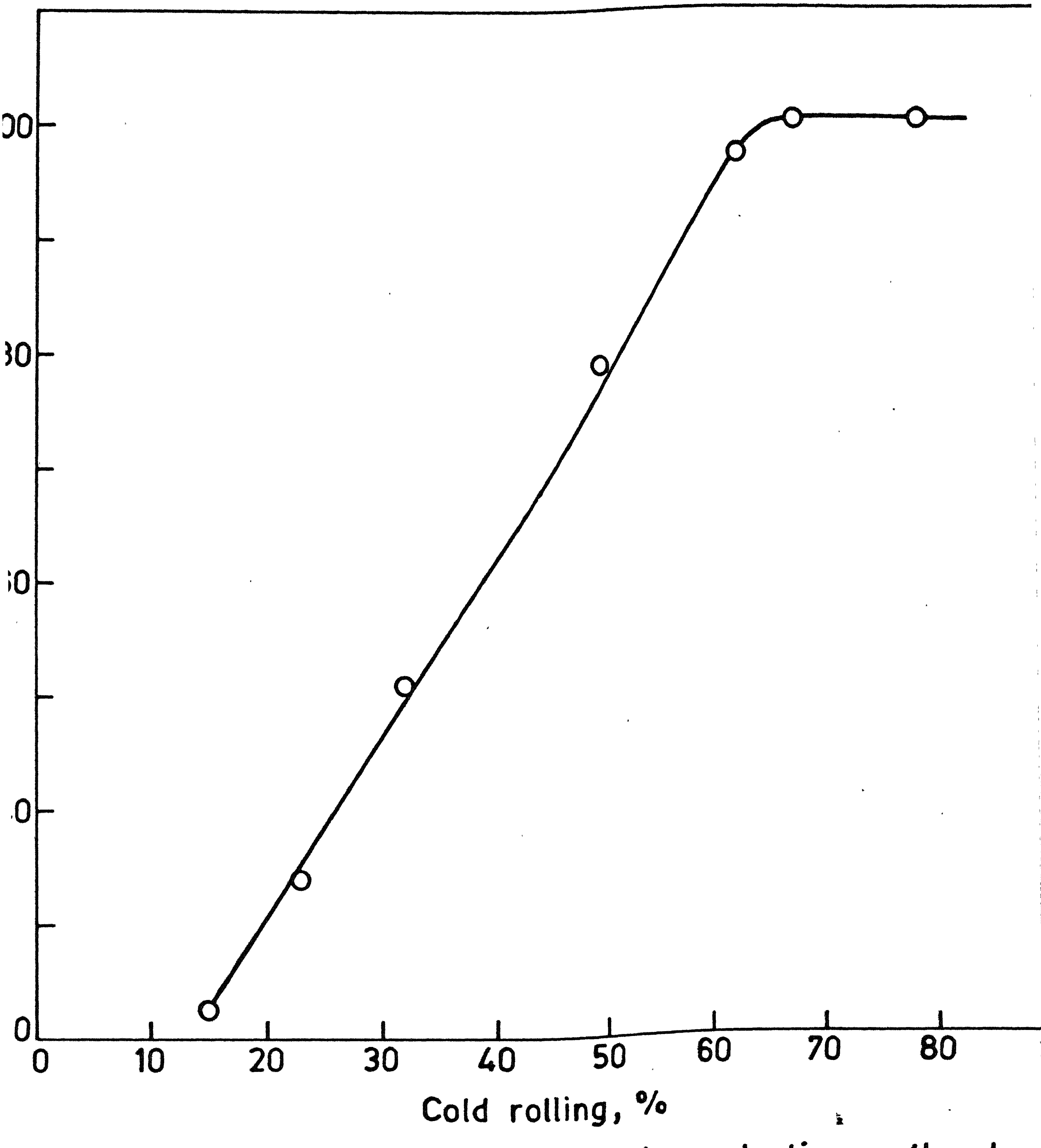


Fig. 3.4 Effect of percentage cold rolling reduction on the densi-
fication efficiency of the sponge iron strip.

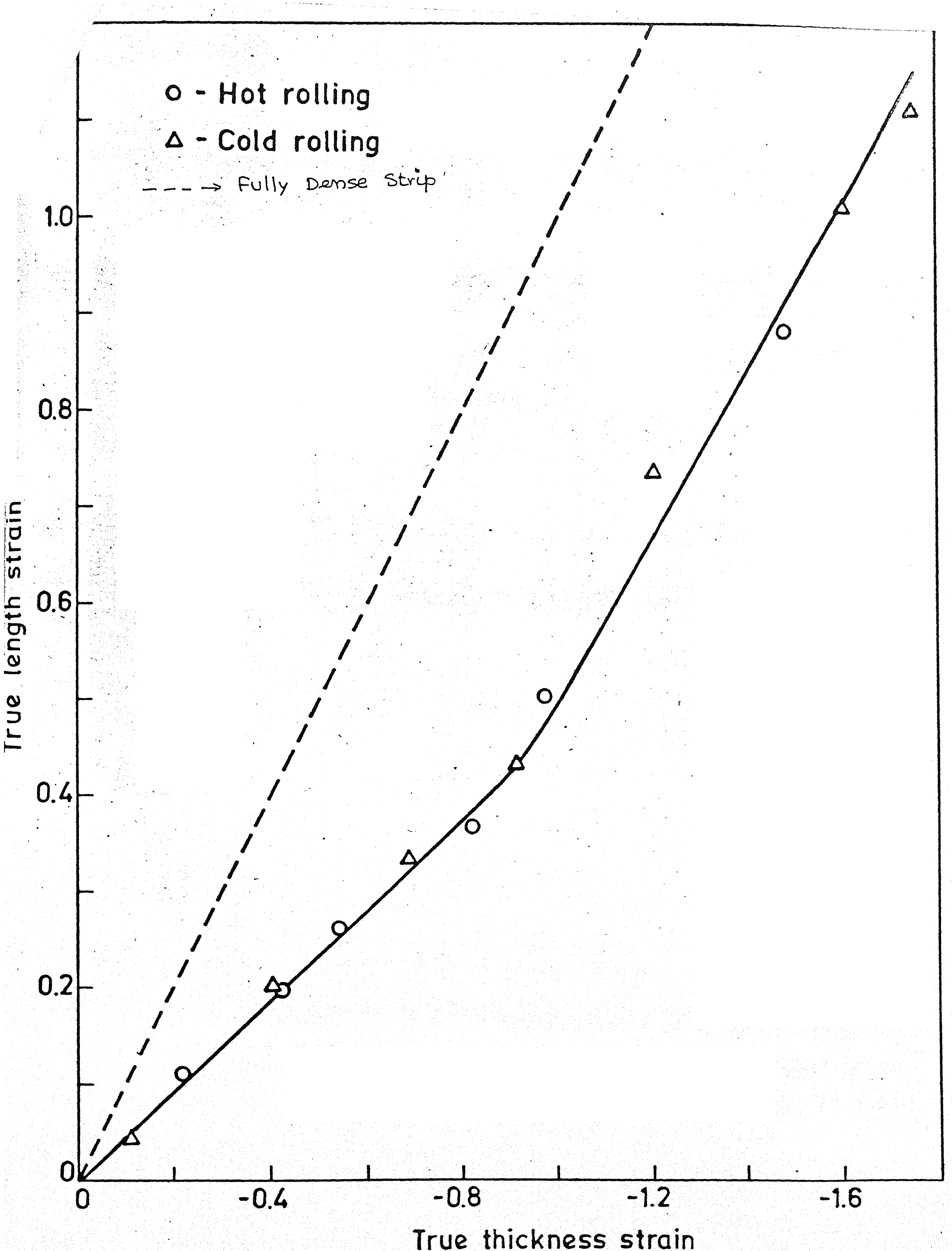


Fig. 3.5 Effect of true thickness strain on the true length strain

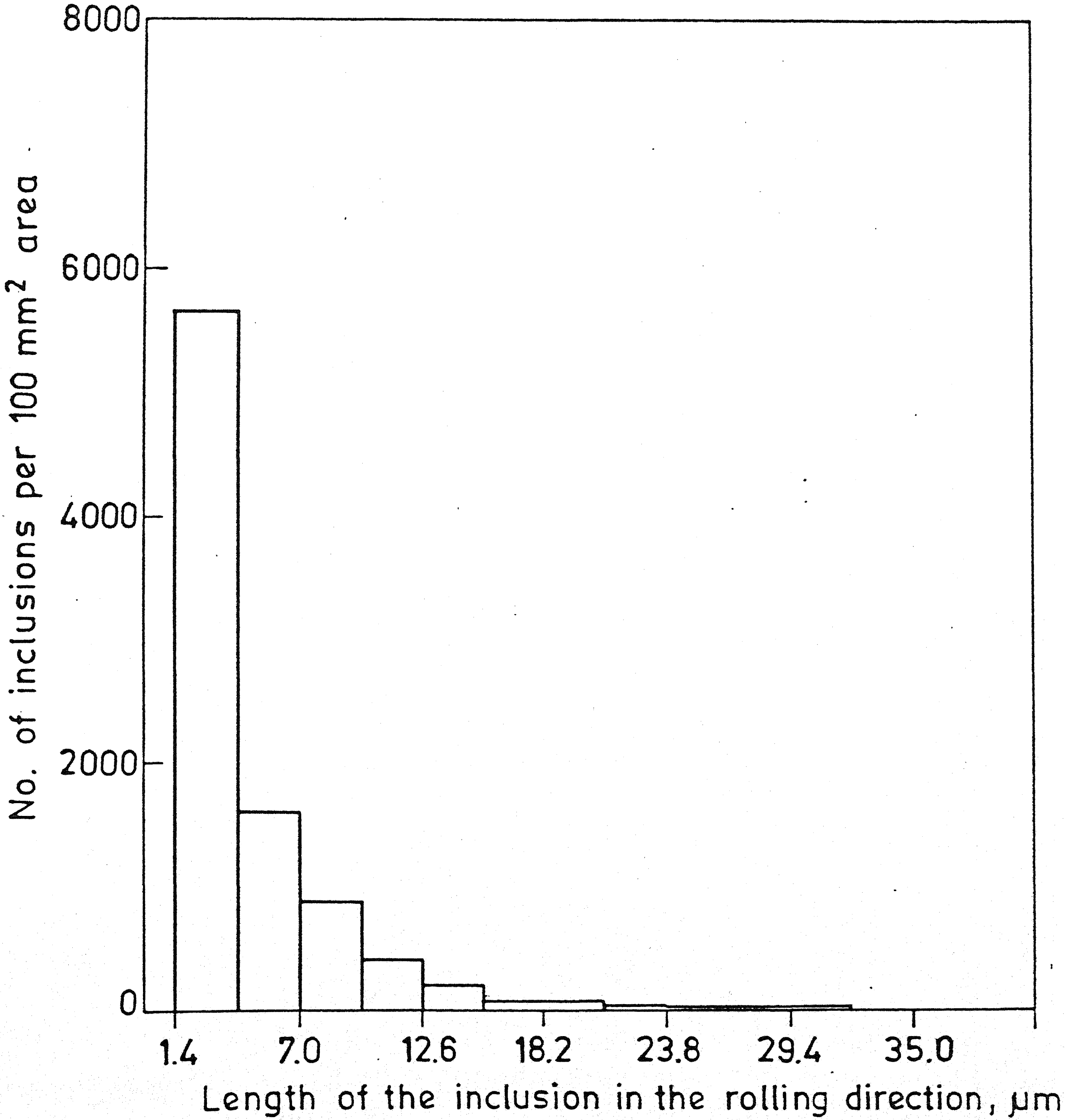


Fig. 3.6 Size distribution of inclusions present in the finished sponge iron strip produced by cold rolling-annealing route.

Table 3.4

Effect of Percentage cold rolling on the densification efficiency of the reduction sponge iron strip.

S.No.	Cold Rolling	Densification Efficiency (%)
1	15	22.5
2	23	34.1
3	32	50.7
4	49	78.6
5	62	97.1
6	67	100
7	78	100

Table 3.5: Effect of true thickness strain on true length strain of the sponge iron strip during cold rolling.

Sl.No.	Percentage cold rolling	Dimensions of the strip before cold rolling x_o, y_o, z_o mm	Dimensions of the strip after cold rolling x_f, y_f, z_f mm	$E\varepsilon_x = \ln \frac{x_f}{x_o}$	$\varepsilon_y = \ln \frac{y_f}{y_o}$	$\varepsilon_z = \ln \frac{z_f}{z_o}$
1.	10	1.85, 55, 79.5	1.66, 55, 82.5	- 0.1083	0	0.0370
2.	30	1.85, 55, 79.5	1.22, 55, 98	- 0.4163	0	0.2092
3.	40	1.85, 55, 79.5	1.08, 55.5, 103.5	- 0.5382	0.0090	0.2638
4.	50	1.85, 55, 79.5	0.92, 56, 110.5	- 0.6985	0.0180	0.3292
5.	60	1.85, 55, 79.5	0.74, 56, 122.5	- 0.9163	0.0180	0.4323
6.	70	1.85, 55, 79.5	0.55, 57, 166	- 1.2130	0.0350	0.7362
7.	80	1.85, 55, 79.5	0.37, 57, 233	- 1.6090	0.0350	1.0753
8.	82	1.85, 55, 79.5	0.32, 57, 267	- 1.7546	0.0350	1.2115

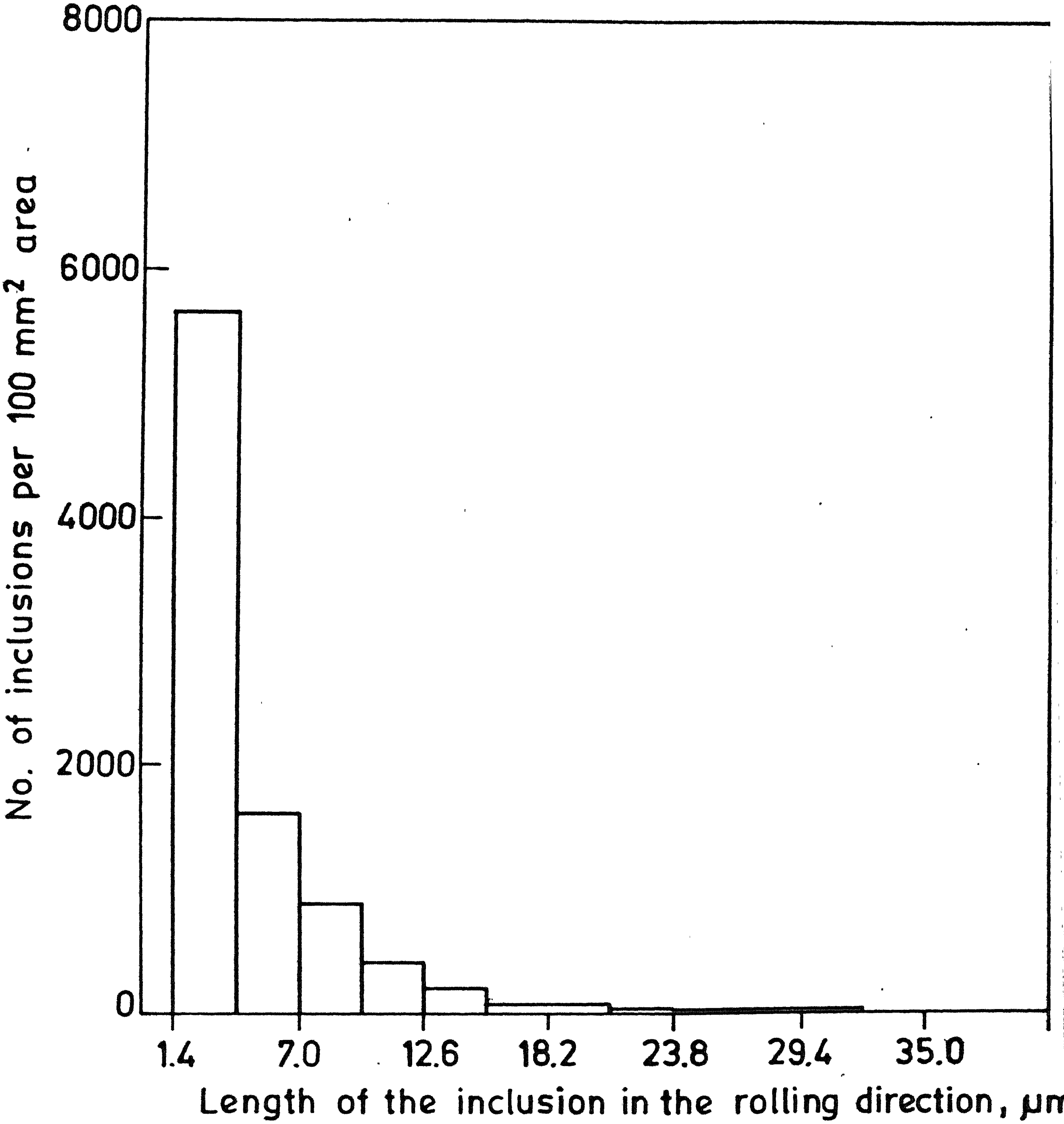


Fig. 3.6 Size distribution of inclusions present in the finished sponge iron strip produced by cold rolling-annealing route.

Table 3.6

Size distribution of inclusions present in the finished sponge iron strip produced by cold rolling - annealing route.

Length of the inclusion in rolling direction m	No. of inclusions per 100 mm^2 area
1.4 - 4.2	5662
4.2 - 7.0	1814
7.0 - 9.8	870
9.8 - 12.6	389
12.6 - 15.4	183
15.4 - 18.2	46
18.2 - 21.0	46
21.0 - 23.8	23
23.8 - 26.6	0
26.6 - 29.4	23
29.4 - 32.2	23
32.2 - 35.0	0
35	23
> 35	0

Table 3.7

Number of inclusions greater than or equal to x in length present in cold rolled annealed finished sponge iron strip and hot rolled - cold rolled - annealed finished strip.

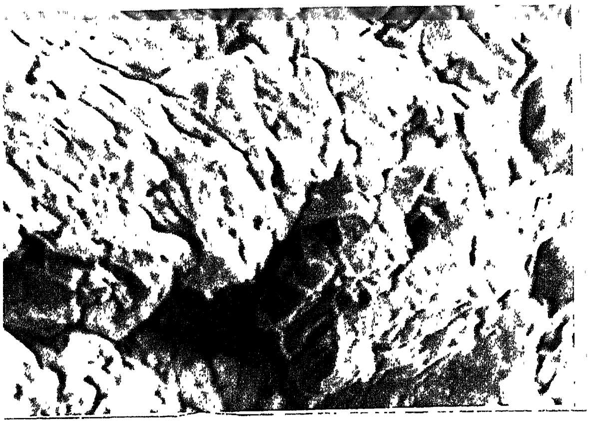
Length of the inclusion in the rolling direction (x) μm	No. of inclusions equal to greater than x in length per 100 mm^2 area in cold rolled annealed samples	Length of the inclusion in the rolling direction (x) μm	No. of inclusions equal to greater than x in length per 100 mm^2 area in hot rolled-cold rolled - annealed samples.
1.4	9102	1.4	11431
4.2	3440	4.2	2938
7.0	1626	7.0	1378
9.8	756	9.8	567
12.6	367	12.6	284
15.4	184	15.4	142
18.2	138	18.2	101
21.0	92	21.0	40
23.8	69	23.8	20
26.6	69	> 23.8	0
29.4	46		
32.2	23		
35	23		
> 35	0		

size distribution of inclusions present in the cold rolled - annealed sample. The minimum size which could be detected by the optical image analyser was 1.4 µm. The size range of the inclusion was found to be 1.4 - ~~3.8~~ 35 µm. However, most of the inclusions were in the range 1.4 - 9.8 µm, and the proportion of inclusions greater than 9.8 µm was rather small. It is quite likely that there are some inclusions finer than 1.4 m which have not been detected optically in the study.

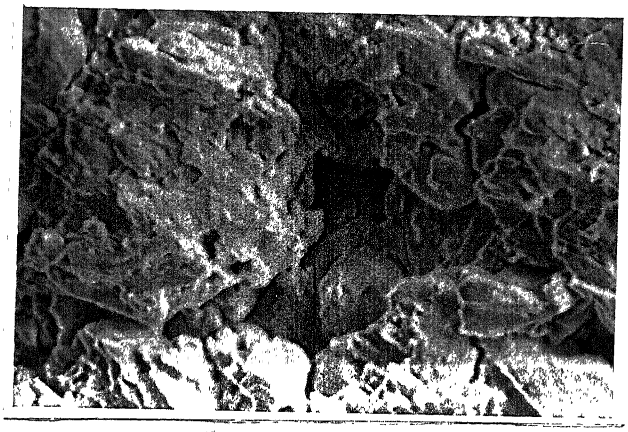
3.1.6 <u>Microstructures</u>:

Fig. 3.7 shows the scanning electron micrograph of the fractured surface of the cold rolled - annealed sponge iron strip at different percentage cold rolling reduction. It can be seen from Figs. 3.7(a) and (b), which are for strips cold rolled to 12% and 23% respectively, that there is no elongation of particles in the rolling direction. It can be seen from Fig. 3.7(c) that particles have started elongating when cold rolled to 35% (area A). The fractured surface of the strip cold rolled to 53% (Fig. 3.7(d)) shows typical dimple type of structure, suggesting that full densification has taken place at various areas although the strip as a whole did not densify fully. Fig. 3.7(e) shows the fractured surface of the strip cold rolled to 72% at which the strip has been fully densified and typical dimple structure was observed.

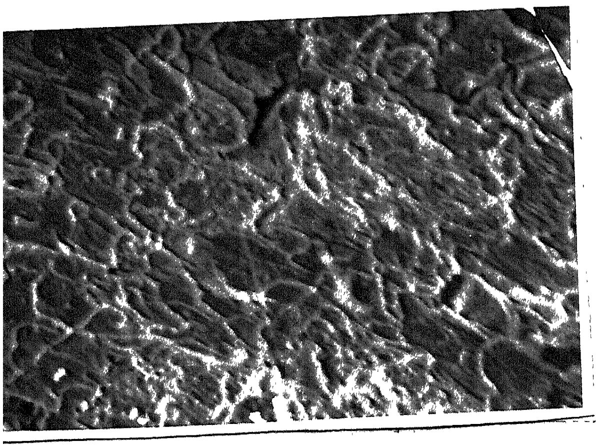
Fig. 3.8(a) shows optical micrograph of the fully dense cold rolled strip annealed at 973 K for 8100 secs. in etched



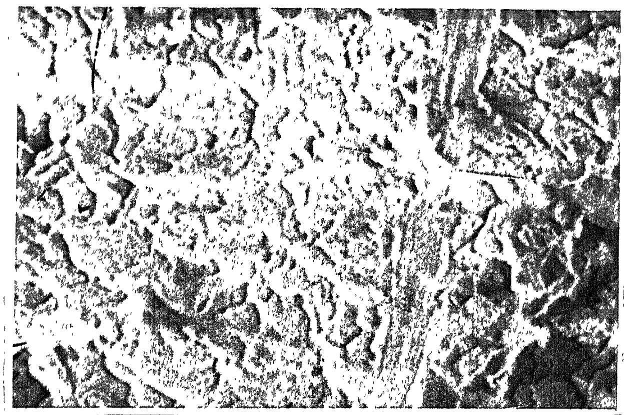
(a)



(b)



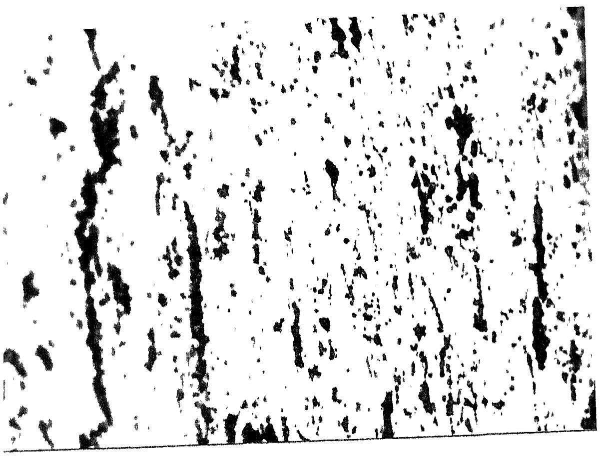
(d)



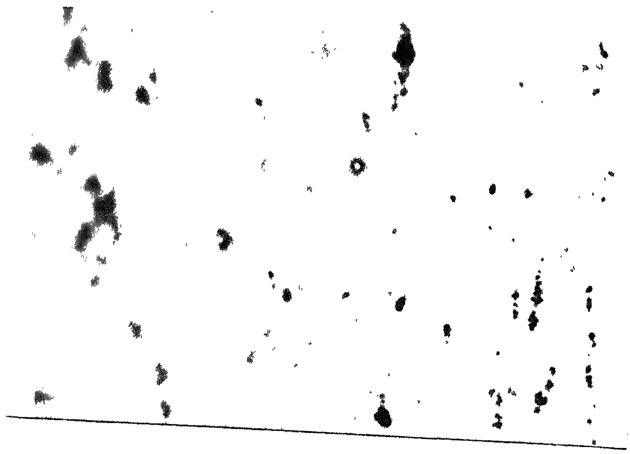
(e)

Fig. 3.7: SEM view of the fractured surface of cold rolled - annealed sponge iron strip.

a) 12 CR (X 250) b) 23 CR (X 250)
c) 35 CR (X 250) d) 53 CR (X 250)



(a)



(b)

Fig. 3.8: Optical micrograph of the fully dense strip obtained by cold rolling - sintering/annealing cycle route.

a) etched condition (X 500)

b) unetched condition (X 200)

condition. Fig. 3.8(b) shows the optical micrograph of the fully dense cold rolled-annealed strip in unetched condition. It shows the non-metallic inclusions present in the cold rolled-annealed strip.

3.1.7 Discussion:

From the foregoing results, it can be deduced that in the initial stages of cold rolling, say upto 30 %, the powder particles are brought closer together in the thickness direction and there is not much elongation of particles in the rolling direction as evident from Figs. 3.7 (a) and (b). Since there is a little increase in the strength in the early stages of cold rolling for both unannealed and annealed specimen, both the strain hardening induced in the particles due to cold rolling and the geometrical hardening brought about in the strip due to the closer proximity of the particles and more area of contacts, are of little significance.

Subsequently, say beyond 30 % cold rolling reduction, the particle starts elongating as can be seen in Fig. 3.7 (c). Both the strain hardening and geometrical hardening become predominant, and the UTS of the strip, measured without any annealing, increased rapidly. However, in the case of UTS of the strip measured after annealing, the increase in UTS is solely due to geometrical hardening.

Similarly, the increase in % elongation of the strip after annealing as a function of cold rolling reduction is solely due to geometrical hardening. However, in the initial stages, say upto 40 % cold rolling reduction, the powder particles are brought closer together in the thickness direction, and there is little change in the elongation values. Beyond this value the particles started elongating, thereby increasing the contact area and the % elongation increased rapidly.

3.2 Densification by Hot Rolling Route:

3.2.1 Effect of Percentage Hot Rolling Reduction on the Density of the Strip:

The relation between density of thestrip and percentage hot rolling reduction is shown in Fig. 3.9 and Table 3.8. As in the case of cold rolling, the density increases linearly with % hot rolling reduction, till it reaches the maximum of 7.76 gm/cm^3 after which the density remains constant. The full density of the strip was obtained at about 60 % hot rolling.

3.2.2 Effect of Percentage Hot Rolling Reduction on the Mechanical Properties of the Strip:

The effect of percentage hot rolling reduction on the UTS and % elongation of the strip, measured after annealing at 973 K for 3600 secs. is shown in Fig. 3.10 and Tab.e 3.9. It can be seen that as the % hot rolling reduction increases, the UTS and elongation increases at a slow rate till 25 % hot rolling reduction and then increases rapidly upto 60 %. Beyond 60

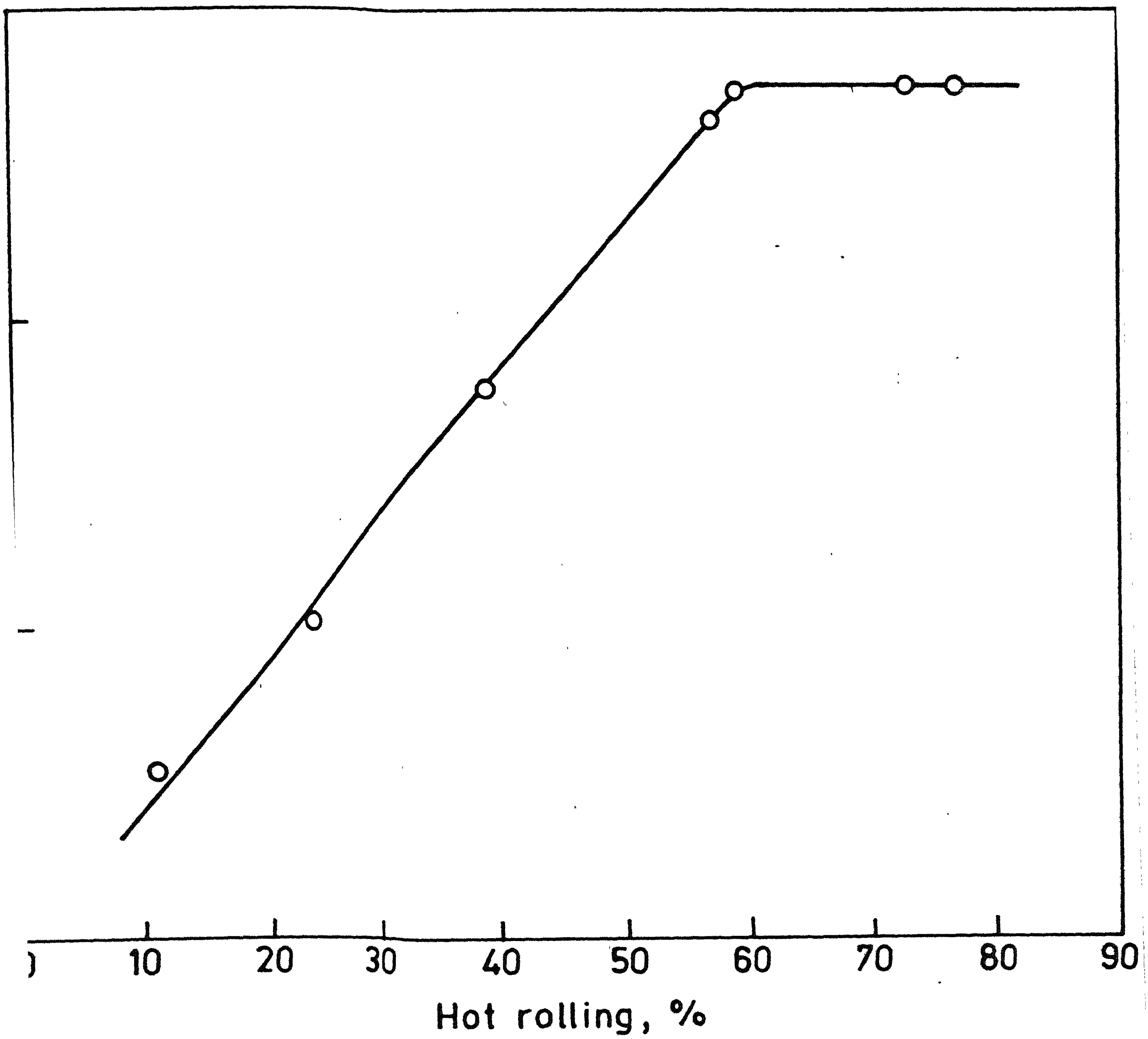


Fig. 3.9 Effect of hot rolling reduction on the density of the sponge iron strip.

Table 3.8

Effect of percentage hot rolling reduction on the density of the sponge iron strip.

S.No.	Hot Rolling (%)	Density gm/cm^3
1	11	5.54
2	24	6.02
3	39	6.78
4	57	7.64
5	59	7.75
6	73	7.76
7	77	7.76

Table 3.9

Effect of percentage hot rolling reduction on the UTS and elongation of the sponge iron strip.

S.No.	Hot Rolling (%)	UTS MN/m^2	Elongation (%)
1	11	82	1.8
2	24	92	3.4
3	39	187	15.6
4	57	246	33.8
5	59	261	33.6
6	73	204	27.5
7	77	175	23.0

Table 3.10: Effect of percentage cold rolling reduction on the UTS and elongation of the finished hot rolled, cold rolled and annealed sponge iron strip.

Annealing temp: 973 °K
Time : 8100 Sec

S.No.	Cold rolling reduction (%)	UTS MN/m^2	Elongation (%)
1	16	255	34.0
2	37	257	35.3
3	60	261	38.0
4	69	235	30.0

hot rolling reduction, a decrease in UTS and % elongation was observed.

3.2.3 Effect of Percentage Cold Rolling on the Fully Dense Hot Rolled Strip:

Fig. 3.11 and Table 3.10 shows the effect of percentage cold rolling on th UTS and % elongation, measured after annealing at 973 K for 8100 secs. ~~on the percentage cold rolling reduction.~~ It can be seen that the UTS increases very slowly upto 60% cold rolling reduction, after which the decrease in UTS was observed. However, the % elongation increases significantly with percentage cold rolling reduction till 60%. Beyond 60%, the decrease in % elongation was observed.

3.2.4 Effect of Percentage Hot Rolling on the Densification Efficiency of the Strip:

The efficiency of densification of the strip by hot rolling, E, was calculated by using the relation given below.

$$E = \frac{(\rho_{HR} - \rho_o)}{(\rho - \rho_o)} \times 100$$

ρ_{HR} - Density of the strip after hot rolling.

ρ_o - Density of the Compacted ~~initial~~ strip.

ρ - Density of the fully dense strip.

Fig. 3.9 shows that the maximum achievable density, in the case of hot rolling, has also been taken as 7.76 g/cm^3 as explained in Sec. 3.1.3. So this value was taken as the theoretical density of the fully dense strip for densification efficiency calculation in the present work.

Variation of densification efficiency with percentage hot rolling reduction is shown in Fig. 3.12 and Table 3.11. It can be seen that the densification efficiency increases linearly with percentage hot rolling till it reaches the limit, after which it remains constant. The maximum densification efficiency was observed at 60 % hot rolling reduction.

3.2.5 Effect of True Thickness Strain on the True Length Strain of the Strip:

Table 3.12 and Fig. 3.5 show the relationship between true thickness strain and true length strain of the hot rolled sponge iron strip. The relationship between the two is the same as that obtained in the case of cold-rolled samples, an explanation of which is given in Section 3.1.4.

3.2.6 Volume Percentage and Size Distribution of Inclusions in the Strip:

The volume fraction of inclusions present in the sample alongwith 95 % confidence limit was found to be 2.93 $\pm$ 0.24 per-cent. The Figs. 3.13 and 3.14 and Tables 3.13 and 3.7 shows the size distribution of inclusions present in the hot rolled to full density - cold rolled - annealed at 973 K for 8100 secs. sample, The size range of the inclusion was found to be in the range 1.4 - 23.8 μm. Though the size range of inclusion was 1.4 - 23.8 μm, most of the inclusions were present in the range 1.4 - 9.8 μm. There might have been some inclusions finer than 1.4 m which could not be detected optically in the present study.

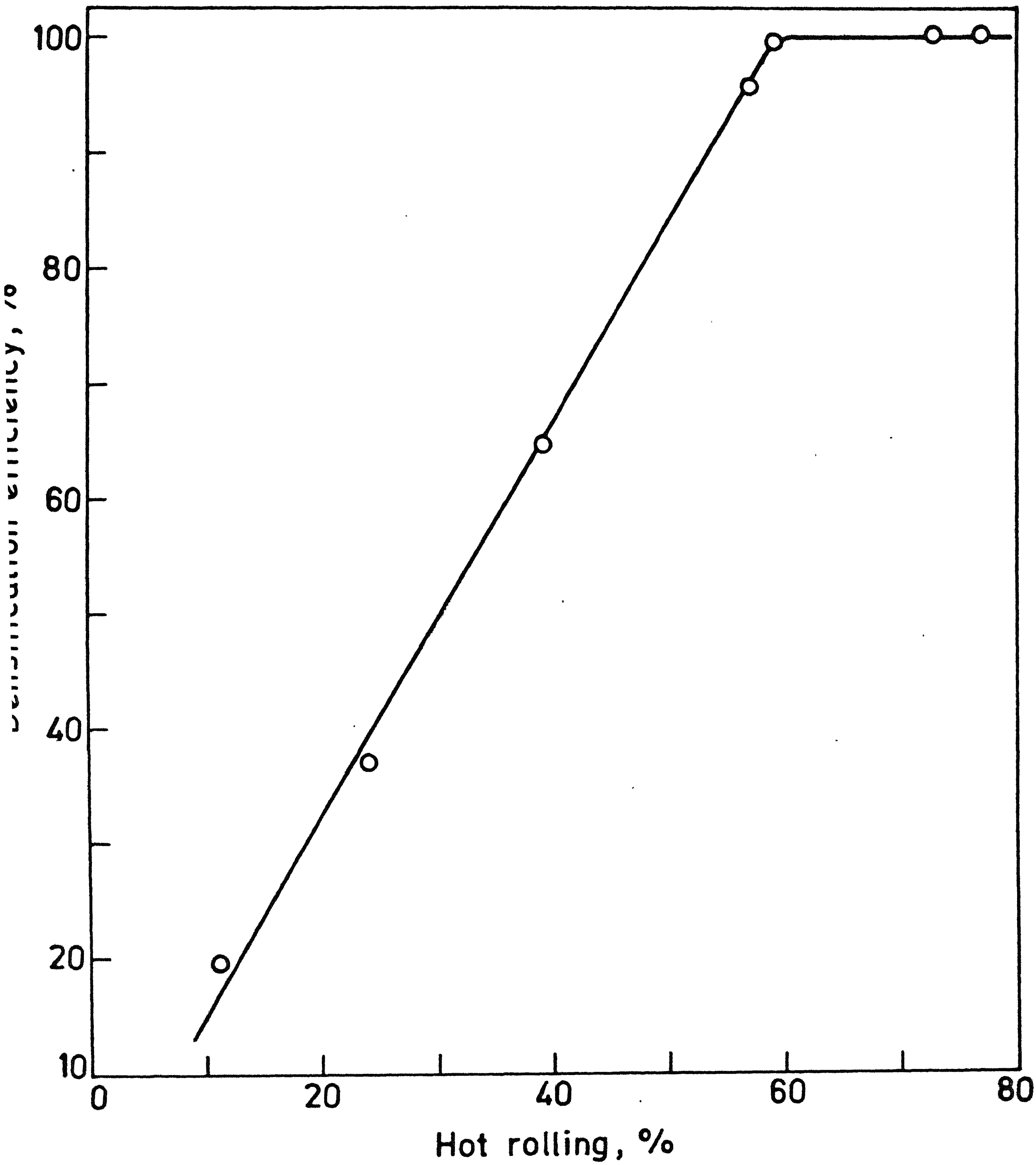


Fig. 3.12 Effect of percentage hot rolling reduction on the densification efficiency of the sponge iron strip.

Table 3.11

Effect of percentage hot rolling reduction on the densification efficiency of the sponge iron strip.

S.No.	Hot Rolling (%)	Densification Efficiency (%)
1	11	19.6
2	24	37.0
3	39	64.5
4	57	95.7
5	59	99.6
6	73	100
7	77	100

Table 3.12: Effect of true thickness strain on length strain of the sponge iron strip during hot rolling.

S.No.	Percentage Hot rolling reduction	Dimensions of the strip before hot rolling x_o, y_o, z_o mm	Dimensions of the strip after hot rolling x_f, y_f, z_f mm	$\varepsilon_x = \ln \frac{x_f}{x_o}$	$\varepsilon_y = \ln \frac{y_f}{y_o}$	$\varepsilon_z = \ln \frac{x_z}{x_o}$
1.	19	1.90, 55, 75	1.54, 55, 84	- 0.2100	0	0.1133
2.	34	1.89, 45, 71.5	1.24, 45.5, 87	- 0.4134	0.0110	0.1962
3.	42	1.80, 61.5, 74	1.04, 62.5, 96	- 0.5485	0.0160	0.2602
4.	57	1.90, 49, 67	0.84, 49.5, 96.5	- 0.8162	0.0110	0.3650
5.	77	1.80, 57.5, 78.5	0.43, 58.5, 190	- 1.4317	0.0172	0.8840

Table 3.13

Size distribution of inclusions present in the finished sponge iron strip produced by hot rolling - cold rolling - annealing route.

Length of the inclusion in the rolling direction	No. of inclusions per 100 mm^2 area
1.4 - 4.2	8493
4.2 - 7.0	1560
7.0 - 9.8	811
9.8 - 12.6	283
12.6 - 15.4	142
15.4 - 18.2	41
18.2 - 21.0	61
21.0 - 23.8	20
23.8	20
> 23.8	0

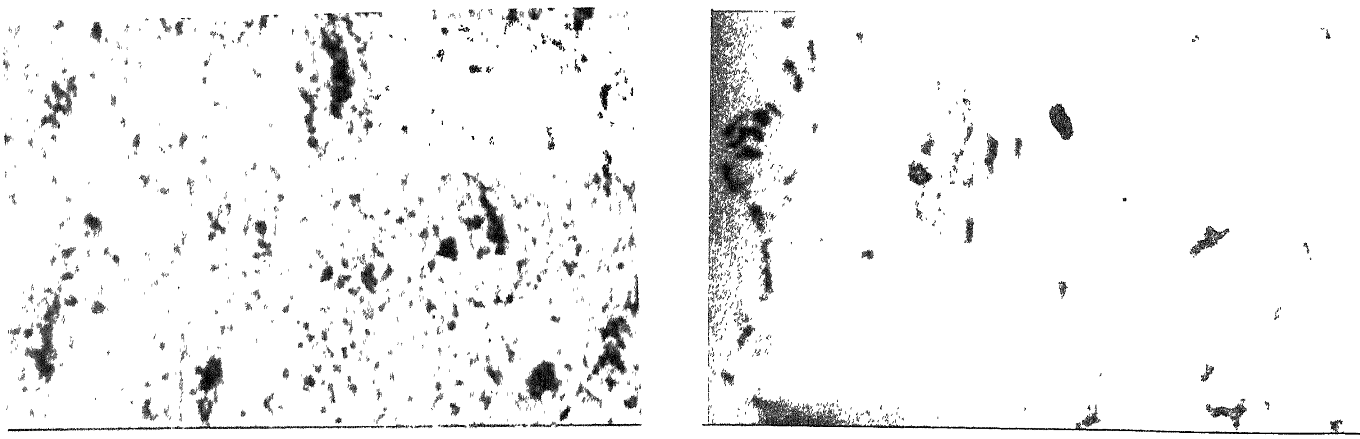
3.2.7 Microstructures:

Fig. 3.15(a) shows the optical micrograph of the hot rolled - cold rolled - annealed finished strip in etched condition. It can be seen from the Fig. 3.15 (a) that, the grain size after cold rolling is small, which is due to the presence of fine inclusions in the strip helping in retarding the grain growth during recrystallisation. Fig. 3.15 (b) shows the microstructure of the hot rolled - cold rolled - annealed strip in unetched condition, showing non-metallic inclusions present in the strip.

3.2.8 Discussion:

During the initial stages of hot rolling, upto 25 % , the UTS and elongation remain almost same. This is because, though a physical movement of the particles in the thickness direction occurs. There is no significant increase in the area of contact between adjacent particles. Beyond 25% hot rolling, the strip undergoes geometrical hardening because of the movement of the particles and area of contact also increases considerably. This results in an increase in the UTS and elongation of the strip.

As the cold rolling of the finished hot rolled strip increases, an improvement in the UTS and the elongation of the strip was observed upto 60% cold rolling possibly due to decrease in grain size. After 60 % cold rolling, the mechanical properties are deteriorating. The present material, infact,



(a) (b)

Fig. 3.15: Optical micrograph of the fully dense strip obtained by hot rolling (60%) - cold rolling (60%) - annealing route

(a) etched condition (X500)

(b) unetched condition (x 200)

is a two phase material consisting of iron matrix and hard non-metallic inclusions dispersed uniformly in the matrix. The plastic behaviour of these two materials are different at room temperature. The shape of these inclusions would not change if the cold rolling reduction is small. However, at high cold rolling reductions, the microcrack would appear at the inclusion-matrix interface. If the cold rolling reduction is too large, the inclusions may also break into smaller size . It seems quite likely that beyond 60% cold rolling, microcrack start appearing at the inclusion - matrix interface, leading to deterioration in the mechanical properties. Electron microscopic studies are needed to confirm the above possible cause of the deterioration of mechanical properties after 60% cold rolling.

3.3 Comparison of Mechanical Properties of the Strips Obtained by P/M Routes and Conventional Route:

Table 3.14 shows the mechanical properties of the strips obtained with different P/M route - iron powder combinations. Properties of the strip obtained by the conventional route is also included.

The mechanical properties of the strips obtained in the present study are shown in Table 3.14 (Sl.No. 8 and 9). It can be seen from Table 3.14 that the UTS and elongation of the strip obtained in the present study by hot rolling - cold rolling - annealing route is slightly better than the strip obtained from cold rolling - annealing cycle route.

Table 3.14: Mechanical properties of the strips produced by conventional routes, and powder metallurgical routes.

S.No.	Powder	Processing route	Apparent density of the finished strip Mg m-3	Yield stress MN/m2	Ultimate tensile strength MN/m2	Elongation %	Ref.
1.	HVA Star electrolytic Iron	Direct cold powder rolling-sintering at 1373 K for 1 hour in H_2 → 10% cold rolling - sintering at 1373 for 1 hr. in H_2 → 30% cold rolling - annealing at 973 K for 1 hr. in vacuum.	7.86	210	247	42	27
2.	Hoganas MH 100.24 Iron	Direct cold powder rolling-sintering at 1373 K for 1 hr. in H_2 → 10% cold rolling - sintering at 1373 K for 1 hr in H_2 → 30% cold rolling - annealing at 973 K for 1 hr. in vacuum.	7.66	177	251	39	27
3.	Hoganas MH 300P Iron	Direct cold powder rolling-sintering at 1373 K for 1 hr. in H_2 → 10% cold rolling - sintering at 1373 K for 1 hr. in H_2 → 30% cold rolling - annealing at 973 K for 1 hr. in vacuum.	7.63	185	259	42	27
4.	HVA Star electrolytic Iron	Direct cold powder rolling (93% dense) -sintering at 1423 K to 1473 K - hot rolling - cold rolling - annealing at 973 K for 1 hr. in vacuum.	7.86	222	249	43	33
5.	Hoganas MH 100.24 Iron	Direct cold powder rolling (93% dense) -sintering at 1423 K to 1473 K - hot rolling - cold rolling - annealing at 973 K for 1 hr. in vacuum.	7.17	117	250	32	33

Table 3.14 continued...

S.No.	Powder	Processing route	Apparent density Mg m^{-3}	Yield stress MN/m^2	Ultimate tensile strength MN/m^2	Elongation %	Ref.
6.	Magnetite super-concentrate powder	Magnetite-super concentrate powder - slurry making → strip making → combined reduction and sintering → hot rolling → 50% cold rolling → annealing at 973 K for 90 min in H_2. (Direct Strip Process)	-	150	309	35	14
7.	Hoganas sponge iron powder (NC 100.24)	Sponge iron powder-slurry making - strip making - pressing - sintering at 1423 K for 1/2 hr. in H_2 → cold rolling → sintering → cold rolling → annealing at 973 K for 2 1/4 hr. in H_2	7.76	-	250	35	Present work
8.	Hoganas sponge iron powder (NC 100.24)	Sponge iron powder - slurry making → Strip making → pressing . sintering at 1423 K for 1/2 hr. in H_2 → hot rolling → cold rolling → annealing at 973 K for 2 1/4 hr. in H_2	7.76	-	261	38	Present work
9.	-	0.2% carbon mild steel strip produced from Blast furnace - Basic oxygen steel making - hot rolling - cold rolling - annealing at 973 K, 90 min. in H_2.	7.86	205	298	41	14
10.	-	IS 513 - 1973 specification for cold rolled and annealed mild steel strip containing 0.15% carbon, 0.06% sulphur, 0.06% phosphorus and 0.5% manganese.	-	-	275	-	39

Serial Nos. 1 and 4 of Table 3.14 shows the mechanical properties of the strip obtained from HVA electrolytic iron powder by repeated cold rolling and annealing, and hot rolling - cold rolling - annealing routes. In this case, there is no significant difference in mechanical properties of the strip produced from these two routes. This result shows that the processing route does not have any effect on mechanical properties of the strip obtained from HVA electrolytic iron powder.

Sl. Nos. 2 and 5 of Table 3.14 gives the mechanical properties of the strip obtained from Hoganas MH 100.24 Iron powder by repeated cold rolling annealing, and hot rolling - cold rolling - annealing route. UTS of the strip obtained from these routes also remains same but the elongation obtained from hot rolling route is found to be lower than the repeated cold rolling - annealing route. However, the density of the strips produced from both routes are not the same. The strip produced from the hot rolling route is less denser (density 7.16 gm/cm^3) than that produced from repeated cold rolling - annealing route (density 7.76 gm/cm^3). Hence the difference in the elongation is not due to processingroute, but due to the different values of density.

As a whole, not much difference in mechanical properties are observed in the strips obtained from various P/M routes. It can be seen that mechanical properties of the conventional low carbon mild steel strip are, in general, slightly higher

than those of P/M iron strip prepared in the present study, despite the fact that the structures are entirely different. The conventional strip contains elements such as Si, Mn, C etc. in solid solutions with iron leading to solid solution strengthening. P/M strip produced in the present study contains virtually zero carbon due to very small carbon in the starting powder on one hand, and prolonged processing in hydrogen at higher temperatures on the other hand. However, the P/M strip contains a large proportion of fine non-metallic inclusions distributed throughout the matrix of iron. It would be more realistic to compare the mechanical properties of the present strip with those made from electrolytic iron powder which does not contain any significant amount of non-metallic inclusions. On comparing the mechanical properties, particularly, UTS, with those of P/M iron strip prepared from electrolytic iron powder (Sl.No. 1 and 4 in Table 3.14), it seems that the relative proportion of very fine, medium and coarse inclusions are such that its commulative effect is not able to produce any strengthening over that of pure iron.

3.4 Size Distribution of Inclusions Present in the P/M Strips and Conventional Mild Steel Strips:

Fig. 3.16 shows the size distribution of non-metallic inclusions present in conventional mild steel strips and P/M strips obtained by various powder metallurgy routes. It can be seen that the maximum length of the inclusions present in the rimmed mild steel strip is above 80 μm. However, most of the

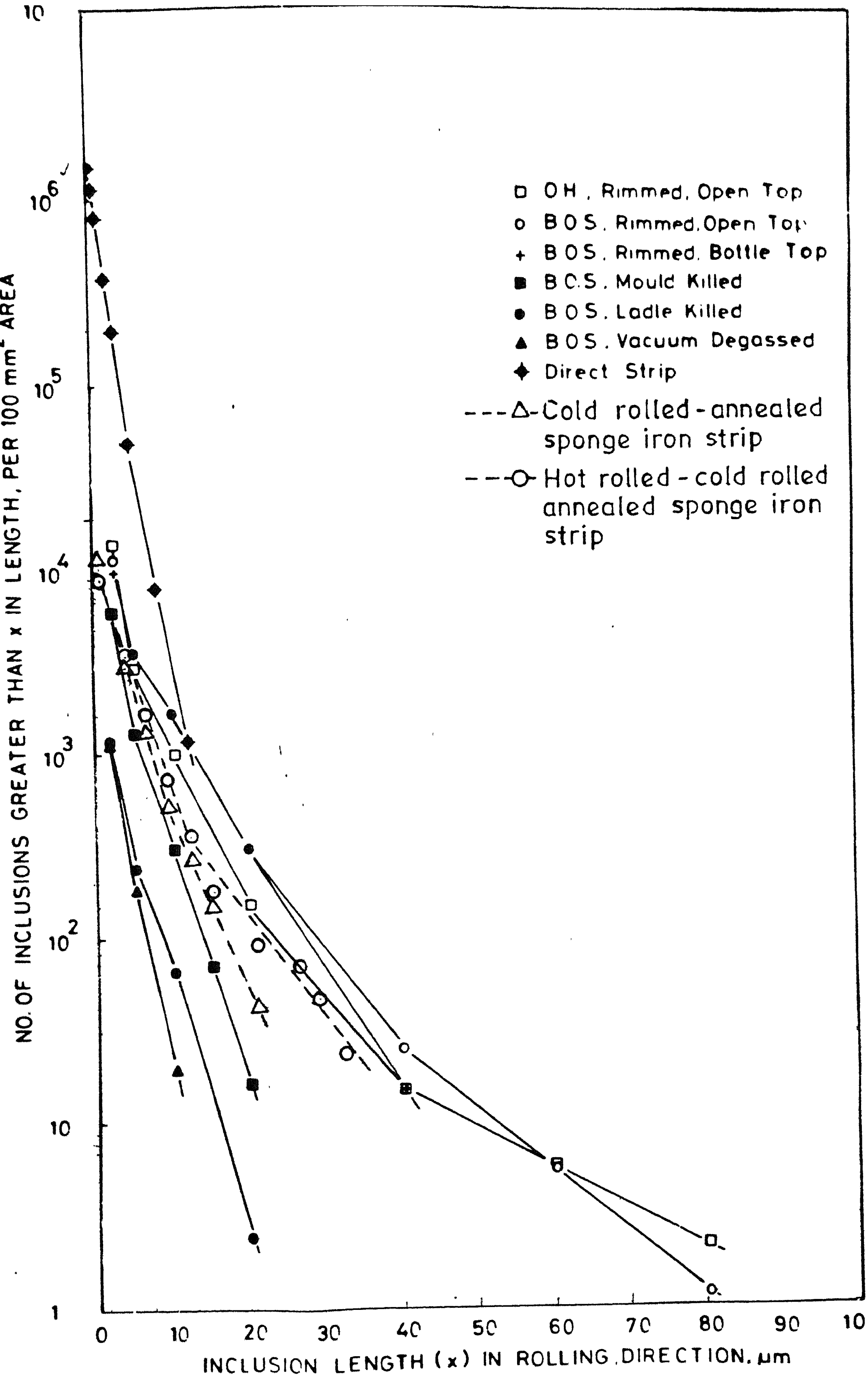


FIG. 3.16 COMPARISON BETWEEN THE CUMULATIVE FREQUENCY / SIZE DISTRIBUTION OF NON-METALLIC INCLUSIONS IN CONVENTIONAL MILD STEEL STRIP, DIRECT STRIP AND P/M STRIP

inclusions present in the range 2 - 40 m. On the other hand, the maximum size of non-metallic inclusion in Al killed steel is abou 20 m, which is considerably lower than that of rimmed steels.The strips obtained direct from iron ore super-concentrate has maximum inclusion size of about 12 m and most of the inclusions are in the range of 0.2 - 4 m.[35] It is clear from the Fig.3.16 that the maximum inclusion size is about 24 m for P/M strip produced from Hoganas iron powder by hot rolling - cold rolling - annealing route, where as it is about 35 m for the strip produced by repeated cold rolling - sintering/annealing route. However, in both the cases, most of the inclusions are in the size range 1.4 to 15-20 m depending on the processing route. It is interest ing to note that inclusions present in Hoaganas SC 100.26 powder has been also in the size range 1 - 30 m, as measured by Quantitative Television Microscope (QTM). The volume percentage of inclusions measured by QTM for the same powder has been reported to be 2.7 , which is very close to our findings on a different but basically same powder.

In rimmed steel only enough deoxidisers such as Fe-Mn are added to maintain the FeO and MnO content within a certain limit. The inclusions present in such steels are mainly oxides of the type (Fe, Mn)O and the sulphides of the type FeS and (Fe, Mn)S. Some inclusions may be of complex compositions consisting of the single phase associated with eutectic mixtures.[36] Some large inclusions comprised of (Fe, Mn)O dendrites in a silicate matrix are also formed.[37]

In general silicates are plastic when rolled in hot working temperature range. MnS inclusions are much more deformable

relative to steel at low temperatures than at high temperatures.[38] For these reasons the maximum size of the inclusions in the rimmed steel as evident from Fig. 3.16 is very large.

In aluminium killed steels, the inclusions may be globular Al_2O_3 clusters or dendritic Al_2O_3 masses. Various calcium aluminates may also be present, which will have melting points depending upon the CaO/Al_2O_3 ratio. Various spinels may also be formed, which will tend to behave similarly to Al_2O_3.[38]

Al_2O_3 is virtually non-deformable at all temperatures, but tends to fracture and form elongated discontinuous stringers during hot working.[38] Al_2O_3:CaO ratio in the calcium aluminates is the key factor in controlling the size of the calcium aluminates. With increasing Al_2O_3:CaO ratio in the calcium aluminates, their particle size decreases. The reason for this is that $CaO.Al_2O_3$, melt at temperature below the ladle temperature, and thus are molten and can coalesce and grow to form larger particles which can float out into the ladle slag. On the other hand, $CaO.2Al_2O_3$ and $CaO.3Al_2O_3$ which are rich in Al_2O_3 are solid and thus form much smaller particles.[37]

No attempt has been made to analyse chemically the inclusions present in the P/M iron strips prepared in the present study. However, an idea about its chemical composition can be had from the composition of the starting material. Hoaganas

powders are produced from high quality magnetite concentrate which contains some irreducible oxides such as Al_2O_3, TiO_2, CaO, SiO_2, etc. These inclusions either present alone or in combinations, are by and large not plastic at working temperatures used in the present study. Moreover, the amount of rolling deformation given to the strip in the present study is rather small. The strip produced from Hoaganas powder, therefore, has smaller size inclusions as compared to conventional strip.

Both the direct strip and P/M iron strip produced in the present study are derived from similar starting material. Direct strip is obtained by reducing magnetite super concentrate (containing ~ 99.3% iron oxide) strip in hydrogen at 1473 K for 1800 secs. During reduction, some sintering between the freshly reduced sponge iron particles are brought about which results into a sponge iron strip. It is subsequently subjected to heavy hot rolling reduction in one pass to produce fully dense strip. The strip is subsequently cold rolled to 50 % thickness reduction and annealed at 973 K for 5400 secs. in hydrogen. On the other hand, the Hoganas iron powder used in the present study is obtained by reducing high quality magnetite concentrate mass kept in a cylindrical shape by coke kept outside and forming a concentric layer with magnetite mass. The layer containing coke also has some limestone to bind any sulphur in the coke and preventing its contamination with the reduced sponge iron. The entire mass is contained in saggers made of

silicon carbide and heated at 1473 K for 24 hours. The reduced iron mass is then ground and subjected to magnetic separation. The resulting powder is finally reduced in hydrogen, re-ground, and annealed.[40]

In the case of direct strips, the total time taken for obtaining sponge iron from magnetite concentrate at 1473 K is much smaller as compared with that taken in making sponge iron powder from magnetite in Hoganas powder manufacturing method. As soon as spinel lattice of magnetite is destroyed during reduction, a new iron lattice is formed and theirreducible oxides, such as Al_2O_3, CaO, SiO_2, TiO_2 etc., which are present in the starting magnetite super-concentrate are precipitated and remains as fine non-metallic inclusion distributed uniformly in the iron matrix. If the material is kept for a longer time at high temperatures, as in Hoganas powder making method, then the non-metallic inclusions will grow in size leading to relatively coarser size and smaller number of inclusions present in the resulting sponge iron powder.

This explains why the strip made from Hoganas iron powder has relatively less in number and coarser in size of non-metallic inclusions than that present in direct strip. It seems likely that this is the probable reason for the higher tensile strength of direct strip as compared with P/M iron strip made from Hoganas iron powder as shown in Table 3.14.

CHAPTER IV

CONCLUSIONS

4.1 Conclusions:

1. The present investigation shows that both the routes, viz. hot rolling - cold rolling - annealing route and repeated cold rolling - annealing route, are capable of producing strips having satisfactory mechanical properties from Hoaganas sponge iron powder. The mechanical properties of the strip produced from both the routes are almost same. It is possible to produce strips having an UTS of 250 -260 MN/m^2, coupled with an elongation of 35 - 38% from sponge iron powder.

2. The volume percentage of inclusion presentin the strip is nearly same in both the routes and its value is ~ 2.93 wt%. However, there is a marginal difference in the maximum size and number of inclusions in both the strips.

3. The amount of rolling deformation required to produce fully dense strip is similar around 60 - 67% for both the routes.

4. The mechanical properties of the strip from Hoganas sponge iron powder (NC 100.24) by both the routes are comparable with the strip obtained from other iron powders such as HVA electrolytic iron powder, sponge iron powder MH 100.24. Although the properties are lower than those of the conventional low carbon mild steel strip used for comparison, but it is close to the specification IS 513 - 1973 for cold rolled and annealed mild steel strip having 0.15 carbon.

4.2 Suggestions for Future Work:

1. In the present work, the UTS and elongation of the strip obtained from sponge iron powder decreases after full densification of the strip. Investigation should be made to ascertain the cause for the behaviour.

2. The mechanical properties has been measured only in the rolling direction. The properties may not be same in all directions. So mechanical anisotropy caused by rolling can be studied.

3. The detailed analysis of the mechanical properties such as plastic strain rate, limiting drawing ratio etc. should be carried out.

4. Electron microscopy study of the strip should be done in order to analyse the very fine inclusions present in the strip.

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